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John A. Myers

Born May 29, 1853; died April 8, 1901.

U. S. DEPARTMENT OF AGRICULTURE.

BUREAU OF CHEMISTRY—BULLETIN NO. 67.

H. W. WILEY, Chief of Bureau.

PROCEEDINGS

OF THE

EIGHTEENTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS

HELD AT

WASHINGTON, D. C.,

November 14, 15, and 16, 1901.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1902.

LETTER OF TRANSMITTAL.

U. S. Department of Agriculture,
Bureau of Chemistry,
Washington, D. C., January 31, 1902.

Sir: I have the honor to transmit to you herewith the manuscript of the proceedings of the eighteenth annual meeting of the Association of Official Agricultural Chemists, with the request that it be published as Bulletin No. 67, of the Bureau of Chemistry.

H. W. Wiley,
Chief of the Bureau of Chemistry and Secretary of the Association of Official Agricultural Chemists.

Hon. James Wilson,
Secretary of Agriculture.

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ORDER OF BUSINESS.

The following order of business was observed:
The president's address.
Reports of the referees in the following order:
First. Report on nitrogen.
Second. Report on potash.
Third. Report on phosphoric acid.
Fourth. Report on soils.
Fifth. Report on ash.
Sixth. Report on foods and feeding stuffs.
Seventh. Report on liquor and food adulteration.
Eighth. Report on dairy products.
Ninth. Report on sugar.
Tenth. Report on tannin.

Eleventh. Report on insecticides.

Twelfth. Reports of special committees (abstract committee, food standards, fertilizer legislation. volumetric standards).

Mr. Van Slyke addressed the convention as follows:

PRESIDENT'S ADDRESS.

Gentlemen of the Association of Official Agricultural Chemists: This is the eighteenth annual convention of our association. To one making a review of the proceedings of the association for these years one striking fact becomes prominent, namely:

THE EXPANSION OF THE WORK.

The original scope of the work of the association was limited to a study of methods relating to the determination of nitrogen, phosphoric acid, and potash in commercial fertilizers. Fifteen years ago a process of evolution began and has continued until to-day we have the original 3 divisions increased to 11. But the actual growth of work is greater than that implied by this statement. since each of the added divisions has involved a group of determinations rather than a single one. For example, the single subject of dairy products is subdivided into three parts—butter, milk, and cheese—while each of these involves several distinct determinations. The subject of liquor and food adulteration has been split up into 15 separate sections. Originally 3 committees took charge of all the work of the association; to-day there are 11 referees and 25 associate referees, not to mention standing committees on food standards. volumetric standards, and fertilizer legislation, which are carrying on additional work.

The primary impulse in the direction of expansion was given by Dr. Wiley in his address as president of the association in 1886, when he said:

> A first step forward has certainly been taken, but the road before us is still a long one. * * * The good results of our meetings might be widely extended if our investigations were made to include a wider range of subjects. Every problem connected with chemical agricultural analysis falling within the range of the studies of anyone connected officially with the agricultural interests of the country seems to me to be a proper theme for our discussion here. Thus, all problems relating to the adulteration of food, the general method of agricultural analysis, and all other matters which concern in common the analyst and the public could be included among the duties and privileges of this body.

This suggestion was at once acted upon and the development of our work followed. We can repeat now with perhaps more emphasis the statement made fifteen years ago: "The road before us is still a long one."

CHANGES IN CHARACTER OF WORK.

During these years there has gradually come a marked change in the character of the chemical work done by the association apart from that involved in its expansion. At first the work was comparatively simple in kind, involving a minimum of research work by individuals and that not especially difficult; but with advance in methods the easier ground has been covered and there is arising a demand for more original investigation, increasingly difficult and complicated in character. Some of our methods of analysis must remain unimproved, unless individual members of the association devote much time to special research. More and more new methods and modifications must be worked out in our individual laboratories and then presented to the association for final testing. To make my meaning more precise, let me illustrate, using the analysis of cheese as an example. The association methods as given at present provide for no determination of the various classes of nitrogen compounds. We know that what we were formerly pleased to call casein in cheese is really a complex mixture made up of many different nitrogen compounds, such as paracasein, nuclein, albumoses, peptones, amides, ammonia, and perhaps others. Something must be done toward isolating and identifying the individual compounds before we can hope to provide effective methods for their estimation, and this involves an immense amount of hard work, which must be done largely by individual initiative. Such work the association can not easily lay out and assign, but until such work is accomplished there is little hope for advancement in the development of methods.

I believe it should be the policy of this association to encourage even more than it has in the past the presentation of papers embodying results of research in all lines bearing in the least upon the study of methods. Sooner or later our work in the perfection of analytical methods will depend entirely upon advanced work of this character. Each year makes this fact more prominent.

Incidentally, in connection with such changes in the character of our work, it may be observed that the agricultural chemist of the future who would be most useful needs a much broader preparation for work than that of a mere analyst. A dozen years ago the demand for agricultural chemists was so great in proportion to the supply that an efficient routine analyst with some experience along agricultural lines was at a premium. Good routine analysts are plentiful now, but there is a growing demand for men who have had a broad and thorough training and possess also the proper temperament for research work. The chemical problems that come to us are constantly growing more complicated and difficult. We are being more and more irresistably drawn into the fascinating but trying field of life chemistry.

A dozen years ago or less scarcely an experiment station chemist would have dared to devote his time to a problem whose results could not be quickly interpreted in dollars and cents to the farmers. But we are gradually getting a little away from such conditions, and some now dare to approach the solution of questions that partake more of the character of advanced science.

FURTHER EXTENSION OF WORK.

The time may not yet be ripe for further expansion in the scope of our work, but it may not be out of place to make a suggestion at this time, looking forward to a desirable extension as soon in the future as may be found practicable. The suggestion I have to make is amply covered by that clause of our constitution which states that one object of the association is "to afford opportunity for the discussion of matters of interest to agricultural chemists." I believe that the time is near at hand when the association should make some effort to encourage and provide for the presentation and discussion at our meetings of papers pertaining to lines of investigations, largely chemical, but not relating necessarily to methods of analysis. Many of our members are carrying on valuable experimental research work in studying chemical problems relating to various lines of agricultural work. Several stations have in hand investigations bearing on some phase of the chemistry of animal nutrition. One station has done much work of interest in studying the relation of lime compounds to plant nutrition, and another has been investigating the influence of various forms of plant food upon the chemical composition of fruits and plants. At least two of our members have done work relating to the manufacture and composition of cider and cider vinegar. Two stations are studying the biochemical problems of cheese ripening, and I might continue for some time naming similar lines of research, but these suffice for illustration. Now, there is really no other place or occasion where these subjects can be so properly presented and discussed as at the meetings of the Association of Official Agricultural Chemists, because only here can they receive thoroughly intelligent and appreciative attention and consideration in the presence of a large body made up of agricultural chemists. Neither in the chemical section of the American Association for the Advancement of Science, the meetings of the American Chemical Society, those of the Society for the Promotion of Agricultural Science, nor in the Association of Agricultural Colleges and Experiment Stations, can those subjects of most interest to us as chemists receive the amount or kind of attention that can be given to them here. I believe some time each year could be devoted to such additions to our progress without unduly prolonging our meetings or taking time from the features that constitute the bulk of our work.

EXPEDITION IN PROCEEDINGS OF CONVENTION.

Permit me to suggest that it is possible for us to expedite our business and economize time without in any degree slighting our work. Undoubtedly we have all noticed opportunities for improvement in this direction during the past. I desire to call attention to a few points in this connection.

First. In too many instances the reports of referees have been presented in forms so incompletely digested as to consume time needlessly. The ideal report of a referee should exclude every detail that is not helpful in enabling members to form an intelligent opinion of the matter presented. Many details may go into the printed report that need not be read on the floor. What we want presented on the floor of our convention are sharp, clear, comprehensive statements of results, with only such detailed explanations as are needed to make those statements readily understood. Too often in the past time has been unprofitably consumed by the referee in reading every figure of every individual worker, even when printed tables containing all the data were in our hands. There has been great improvement in this respect during the past ten years, but a still greater improvement is in order.

Second. If time may not be saved. interest at least will be increased if referees and all speakers will develop enough lung power to let others know what they are saying.

Third. In our discussions it too often happens that old ground which is or ought to be familiar is repeatedly gone over year after year by some speakers, usually by those who have not attended the meetings regularly. It may be safely assumed that the general facts of elementary chemistry do not need repetition here. It would yield beneficial results if all of us would take time now and then to look over the proceedings of previous meetings.

Fourth. Extreme care should be taken in the selection of the chairman of the committee on recommendations of referees. The duties of the place are arduous at best and especially in presenting matters to the convention. which requires a strong voice, system, ability to state facts sharply and clearly, self-possession, an understanding of the facts presented, patience, alertness, and omnipresence. The wrong man in this place can easily waste one or two hours of the convention's time. With increase of work it might be wise to divide this labor among three committees. A plan will probably be brought forward on this and associated points.

Fifth. One other suggestion in connection with saving time is the matter of promptness in attendance at the opening of every session.

Of all the associations with which I have had any experience, this is unquestionably the hardest working. The foregoing suggestions have been made not with the intention of making us work still harder, but in the hope of so conserving our time and efforts that our results may be no less effective with the expenditure of less time and labor.

DEATH OF JOHN A. MYERS.

During the past year there has passed from life one who was from the beginning intimately connected with the work of this association, Mr. John A. Myers, president in 1889. It is highly fittting that a special evening session should be held by us in honor of his memory.

DISCONTINUANCE OF THE COMMITTEE ON ABSTRACTING.

The discontinuance of the committee on abstracting is suggested. as this work is now fully provided for in the Office of Experiment Stations.

CHEMISTS AS PRACTICAL MEN.

In conclusion, I want briefly to consider one more topic, the discussion of which in this place may seem a departure from our traditions, but its justification will appear in due time. The topic is "Chemists as practical men."

I remember of once hearing, at a meeting which I attended, the statement that chemists in general were quite outside the circle of those possessing plain, common, everyday horse sense. It was emphatically stated, with an effort at dramatic eloquence, that the training and habits of life of the chemist led him to such retirement from contact with the world and isolation from his fellow men that he was necessarily and unavoidably deficient as a man of affairs, sadly lacking in executive ability and knowledge of men, etc.—in fact he was pictured as a kind of intellectual babe in the wood when he went outside his laboratory. This was an echo of the old traditional cry that a man of scientific training could not possibly be a practical man in the affairs of everyday life. The conclusion drawn by the speakers was that the chemists could not be expected and should not be allowed to occupy positions calling for executive ability and acquaintance with the ways of men, places requiring the exercise of good judgment and plain common sense. It was also concluded that chemists, having no sense outside the laboratory except that of smell, should not obtrude their impracticable and valueless opinions in public councils. They might be seen, but not heard. Under the circumstances, I thought it best to keep still. It has seemed proper, therefore, that a defense should be entered where the audience would be more fully sympathetic.

I can not too strongly urge you who have not done so to read an article entitled "The man of science in practical affairs," by Dr. F. W.

Clarke, in Popular Science, February, 1900. He discusses the question in a most thorough and convincing manner.

In closing, it may be pertinent to state that more than one-third of the directors of our experiment stations and a few college presidents have been active members of this association. The fact is that if a man has in him the elements of being practical, a chemical training seems peculiarly adapted to develop that side of his personality.

As Dr. Clarke says, in the article referred to:

> At bottom the scientific training is a training in clear, thorough, precise statement, accurate observation, the verification of evidences, and the ascertainment of truth. Why should its recipient be unfitted for practical things? Good administration, the effective transaction of business, implies system, exactness, the judgment of evidence upon its merits, and the prompt solution of problems as they arise, and to each of these requisites the scientific education is directly related. What other training is less likely to produce dreamers or more likely to develop efficient men?

APPOINTMENT OF COMMITTEES.

The President. It has been the custom at this point of the proceedings to appoint committees, and if there is no objection I will announce these appointments:

The committee to wait upon the Secretary and Assistant Secretary of Agriculture, to inform them that we are in session, and that the members of the association will be pleased to have them meet with us, will consist of Messrs. Kilgore and Scovell.

Committee on resolutions: Messrs. Frear, Wheeler, and Jenkins.

Committee on nominations: Messrs. Huston, Woods, and McDonnell.

Committee on amendments to the constitution: Messrs. Wheeler, Caldwell, and Winton.

Mr. Williams. For some years past the association in its preliminary actions has followed the practice of securing careful consideration and expression—the need of which had been previously demonstrated—in addition to that given by the referee, in regard to the appointment of a committee on recommendations. Within the last two or three years our work has branched out in such a manner as to involve the consideration of a much greater number of different classes and kinds of methods than this association has heretofore undertaken to examine, and it appears that a number of the members of the association think that to expect one committee on recommendations to give mature consideration to the recommendations on the large variety of subjects treated is not just to the committee nor to the subjects. I would therefore move, Mr. President, that a committee of three be appointed by the Chair to consider and report to the association at this morning's session how many committees should be appointed to act on the recommendations of referees upon the several classes of subjects.

The PRESIDENT. The motion has been made and seconded that a committee be appointed to report on increasing the number of committees on the recommendations of referees. The motion is carried. I will appoint Messrs. Frear, Wheeler, and Kilgore.

The next item in the regular order of business is the presentation of the report on nitrogen.

Mr. WHEELER. If there is no objection, Mr. President, I should like to introduce a motion.

The PRESIDENT. If there are no objections you may have the floor for that purpose.

Mr. WHEELER. In view of the fact that the number of referees has been increased perhaps two or three times, and the difficulty that any one man has in selecting these referees, I desire to offer an amendment to the constitution. The proposition has been made that the appointment of referees be referred to a select committee rather than to the president of the association. The proposed amendment is as follows:

Resolved, That section 4 of the constitution be amended by substituting for the word "president," in the first line of the section, the words "executive committee."

I move that a committee be appointed to consider this resolution.

Mr. KILGORE. I would call for the reading of that part of the constitution which it is proposed to change.

The PRESIDENT. The member will please read the section as it now is and the correct change which he proposes.

Mr. WHEELER (reading):

(4) There shall be appointed by the president, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

According to the proposed change it would read:

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and associate referee for each of the subjects to be considered by the association.

The PRESIDENT. Has this motion been seconded?

Mr. FREAR. I second the motion.

Mr. KILGORE. What article of the constitution gives authority for amending it?

The PRESIDENT. Section 10.

All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

Are there any remarks upon the motion in regard to the appointment of a committee to which shall be referred the resolution to amend section 4 of the constitution by placing the appointment of referees in the hands of the executive committee? If not, all who are in favor will say "Aye." Opposed, the same. It is carried.

Mr. Frear. I would like to introduce another resolution affecting the constitution, and move that it be referred to the consideration of the same committee. This resolution affects section 7, which at present reads as follows:

> (7) No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

The association has of late years taken up quite a large amount of work affecting the official investigation of the various kinds of trial inspection. It seems to me that the same thing can be said in regard to the soil-inspection work, in which a large number of us are concerned. I would therefore propose, Mr. President, the following:

> *Resolved*, That section 7 of the constitution be amended by substituting, in the first line, for the words "fertilizer analyses," the words "analysis used in official inspection," and in the third line for the words "fertilizer work" the words "the particular inspection affected."

The President. A motion has been made to change the constitution in the manner specified. Are there any remarks?

Mr. Ewell. I would like to suggest that some time limit be inserted. Should it not be a year, or a certain length of time?

The President. That would probably be considered by the committee.

Mr. Winton. Is there anything in the proposed change that would prevent the introduction of additional propositions?

Mr. Frear. Not at all.

The President. This will be referred to the committee on amendments to the constitution, Messrs. Wheeler, Caldwell, and Winton. Mr. Perkins has said that he would have his report here, but it has not yet been received, and the next in order is the report of the referee on potash. Is he present?

Mr. Ross. The referee handed the report to me for presentation to the association and asked me to hand it to the secretary, who requests that I should read the report.

Mr. Eaton. I wish to present to the association a letter from Mr. J. B. Noble, as follows:

State of Connecticut, Dairy Commissioner's Office,
Hartford, November 8, 1901.

Dear Sir: At a meeting of the National Association of State Dairy and Food Commissioners held in Buffalo October 15, 16, and 17, 1901, the following persons were appointed as a committee to represent the association before the National Association of Agricultural Chemists, to be held in Washington: Dr. E. N. Eaton, of Illinois; Dr. R. M. Doolittle, of Michigan; Dr. A. S. Mitchell, of Wisconsin; Dr. William Frear, of Pennsylvania.

Yours, respectfully,

J. B. Noble, *Secretary.*

Dr. E. N. Eaton, *Chicago, Ill.*

The PRESIDENT. We have this letter from Mr. Noble. What action shall be taken on it?

Mr. FREAR. I was made a member of this committee, but have just received my notification and am not prepared to submit a report. I was not in attendance with the committee during the national convention in Buffalo at the time its action was taken, but since I am chairman at present of the committee of this association charged with the consideration of food standards, I would offer this resolution for the consideration of the association:

Resolved, That the Association of Official Agricultural Chemists welcomes the action of the National Association of State Dairy and Food Commissioners in appointing a committee of its members to present to our association its views upon the subject of food standards—the formulation of which has already been undertaken by this association for the guidance of its members—and upon the methods of detection of food adulteration.

The PRESIDENT. You have heard the resolution which has been presented to the association with reference to this matter. What action shall be taken with regard to it?

Mr. EATON. I see that the resolution covers, at least in part, the ground that our association has already gone over in the establishing of standards, and it would at least be open to criticism as to the point of practice to publish the different reports. I do not believe that the question of standards was the only one my associates had in mind. The question of the adoption of food analyses was also brought out. I am considerably interested in this topic, yet I shall be as brief concerning it as possible and take up no more time than necessary. I think that possibly this resolution might be adopted to cover the question of the methods of analyses as well as standards for food products and I am sure the members of the committee will be glad to cooperate with the committee appointed by this association to look after the methods of analyses and standards of food products and concur with them or give any advice their experience may have made them competent to give.

Mr. FREAR. I shall be glad to so change the wording of the resolution in the manner suggested by Mr. Eaton, and to have it cover the subject of food sanitation and also the subject of the methods of analyses for the detection of adulteration.

The PRESIDENT. The motion is upon the adoption of this resolution.

Mr. KILGORE. I don't know that I quite understand the import of this resolution. This association is working especially upon the subject of methods. It has been on that question for some time, and last year enlarged the number of men engaged on it to 15, and this year we have given a large part of our time to that work and there will be presented to this association at least the methods advised. Do I understand that the committee from this association is going to confer with the committee of the other societies with reference to these particular methods?

Mr. Frear. In regard to the resolution I might explain further that my purpose in introducing it was to formally welcome an attitude of cooperation with the movement which has been started through the officials organized under the title of National Association of State Dairy and Food Commissioners, who are in charge of the examination of State laws controlling the various human foods in the several States, but not to indicate the method specifically by which this cooperation should be attained. I hardly thought that such a statement was necessary in the resolution for the reason that every chemist who is appointed by this National Association of State Dairy and Food Commissioners is entitled to membership in this body, and therefore is part of this working organization, and as such would be entitled to present any views to any committee of this body just as any other member is entitled, and, also, coming in a representative capacity, he would be entitled to hearing and representation before final action is taken, and any words uttered by him would be given due weight.

Mr. Wheeler. Is there not a committee on resolutions? If so, I would suggest this matter be referred to that committee.

The President. The matter will be referred to the committee without motion, if there is no objection.

We will now take up the report on potash, prepared by Mr. C. L. Hare.

REPORT ON POTASH.

By C. L. Hare, *Referee.*

In the absence of recommendations from the association the referee planned the work on potash for 1901 so as to include the following investigations:

(1) Continuation of the previous years' inquiries, suggested by Mr. F. B. Carpenter, concerning the probable failure of the Lindo-Gladding method to obtain from mixtures of acid phosphate and potash salts the theoretical amount of potash added.

(2) The use of ammonium chlorid as a possible aid in securing solution of all potash present in such mixtures.

(3) The referee for the year 1899 described briefly a method for the determination of potash based upon the use of barium carbonate for precipitation from neutral solution. Later, he abandoned that method and turned his attention to one suggested by that, i. e., the use of milk of lime as the precipitant. His method has given such excellent results in the laboratory of the Alabama station that the present referee felt justified in offering it to the association for trial. Consequently the method, as described below, was included in the work.

During the spring over thirty circular letters were sent out to chemists who, it was thought, might be interested. Twelve analysts expressed a willingness to cooperate. The following letter of instructions was sent out with each set of samples:

Dear Sir:

I have mailed you to-day two samples for official potash work.

Sample No. 1 is chemically pure potassium chlorid. Sample No. 2 is a mixture of chemically pure potassium chlorid and acid phosphate in the proportion of 5 to 95.

1. Determine potash in No. 1 by the official method.
2. (*a*) Determine water-soluble potash in No. 2 by the official method.

(*b*) Determine water-soluble potash in No. 2 by the official method with the exception that 5 to 10 grams of ammonium chlorid should be added to the weighed sample before making solution.

(*c*) Determine water-soluble potash in No. 2 by the following method: Make the solution according to the official method, and while the solution is still hot add milk of lime until alkaline. Boil for a few minutes, cool, make up to the mark, measure out an aliquot portion, acidify with hydrochloric acid, and evaporate with platinum solution; wash with alcohol and Gladding "wash" as in official method.

3. Make blank determinations on reagents, using amounts required for regular determinations.

In reporting results please state what filter was used for filtering potassium platinic chlorid. If convenient use the Gooch filter.

State also the method by which the asbestos was prepared for use. Report amount of insoluble residue after washing out K_2PtCl_6 with hot water.

Please report all observations that appear to be of interest.

C. L. Hare, *Referee.*

Sample No. 1 was potassium chlorid. Sample No. 2 was prepared by mixing intimately acid phosphate and chemically pure potassium chlorid in the proportion of 95 acid phosphate to 5 potassium chlorid. Reports were received from eight laboratories, representing the work of twelve analysts. The following table shows in detail the results obtained:

Table 1.—*Results of analysis of potassium chlorid and of the mixture of potassium chlorid and acid phosphate.*

Analyst.	Sample No. 1, C. P. KCl.	Sample No. 2, 95 acid phosphate and 5 KCl.		
		Lindo-Gladding method.	Milk-of-lime method.	Lindo-Gladding with NH_4Cl.
J. Q. Burton, Georgia	62.82	3.10	3.29	3.13
	....	3.03	3.39	3.11
	62.82	3.07	3.34	3.12
R. G. Williams, Georgia	....	....	3.19	....
	....	....	3.11	....
	....	....	3.18	....
	....	....	3.16	....
I. D. Sessums, Mississippi	62.60	3.15	3.18	3.39
	62.60	3.14	3.14	3.35
	63.10	3.20	3.19	3.34
	62.80	3.14	3.18	3.37
	....	3.14	3.13	3.33
	....	3.15	3.14	3.31
	....	3.14	3.16	3.34
	....	3.12	3.19	3.32
	62.80	3.15	3.15	3.35
O. W. Knight, Maine	62.96	3.14	3.13	3.18
	62.96	3.13	3.12	3.09
	63.00	....	....	....
	62.97	3.14	3.13	3.14
C. D. Holley, Maine	63.00	3.14	3.13	3.24
	63.00	3.16	3.155	3.21
	63.00	3.15	3.14	3.23
C. H. Jones, Vermont	63.21	3.23	3.11	3.28

TABLE 1.—*Results of analysis of potassium chlorid and of the mixture of potassium chlorid and acid phosphate*—Continued.

Analyst.	Sample No. 1, C. P. KCl.	Sample No. 2, 95 acid phosphate and 5 KCl.		
		Lindo-Gladding method.	Milk-of-lime method.	Lindo-Gladding with NH_4Cl.
C. I. Boyden, Vermont		3.20	3.06	3.29
		3.04	3.12	3.24
		3.12	3.09	3.27
F. B. Carpenter, Virginia	62.90	3.10	3.05	3.14
E. L. Parker, Virginia	62.92	3.08	3.08	3.12
T. R. Gough, Maryland	63.80	3.08	3.17	3.22
	62.20	3.03	3.15	3.24
	63.00			
	63.00	3.05	3.16	3.23
R. F. Hare, New Mexico		3.09	3.07	
		3.07	3.08	3.06
		3.08	3.06	
		3.11	3.04	
		3.09	3.06	3.06
C. L. Hare, Alabama	62.91	3.19	3.24	3.17
		3.16	3.24	3.15
		3.14	3.25	3.14
		3.18	3.26	3.13
		3.09	3.28	3.13
		3.18	3.29	3.09
	62.91	3.16	3.26	3.14
Means	62.95	3.12	3.15	3.19

COMMENTS OF REFEREE.

Careful analyses made in the laboratory of the Alabama station of the acid phosphate used in making the mixture from which Sample No. 2 was drawn showed 0.2 per cent potash. Based on the analysis of the acid phosphate and of the potassium chlorid, the theoretical amount of potash in No. 2 was 3.35 per cent.

On reference to the table it is seen that the official method fails to remove the theoretical amount of potash—the difference between theory and the average of results being 0.23 per cent. Results by this method are within 0.18 per cent of each other, and with such close agreement no satisfactory explanation is at hand as to why results are so far below theory.

While the addition of ammonium chlorid before making solution gives an increase of 0.07 per cent potash, the results reported do not warrant the conclusion that ammonium chlorid may be used with advantage. One disadvantage is that the large amount of ammonia salts left after evaporating the solution with sulphuric acid may cause loss from sputtering.

The referee would direct especial attention to the results obtained by the milk-of-lime method. It will be observed that these results, excluding one determination, agree within 0.21 per cent of each other. The average of results agrees within 0.02 per cent of the average of results obtained by the official method. This speaks highly for a method which is having its first trial in the hands of the majority of the analysts.

While the direct evaporation of the solution with platinum solution leaves a rather large amount of lime salts, none of the analysts have reported trouble in the final washings. In washing this residue, analysts who have little experience with the method are apt to subject it to too vigorous rubbing. Experience teaches that excessive pulverization is unnecessary, and will possibly result in loss of potash, caused by the finely divided potassium platinic chlorid passing through the filter. It is the opinion of the referee that in hands familiar with it the method will remove a slightly higher per cent of potash from mixtures of acid phosphate and potash salts than will the Lindo-Gladding method.

The small amount of manipulation required in the method, the fact that evaporation with sulphuric acid—a fruitful source of error—is avoided, the rapidity of the method, and finally the excellent results obtainable, recommend the method as one which may be brought to a very high degree of usefulness. It seems to be applicable to the class of goods on which it was tried, and experiments conducted at the Alabama station indicate that it can be used as successfully on fertilizers containing organic materials of various kinds.

The following table includes results obtained by the writer by applying the method to fertilizers containing such organic materials as cotton-seed meal, tankage, blood, bones, and tobacco stems. The potash content of the samples was, with one exception, unknown, and in parallel columns is given the per cent of potash by the Lindo-Gladding method.

TABLE 2.—*Results on fertilizers containing organic materials.*

Sample.	Lindo-Gladding method.	Milk-of-lime method.
1	2.55	2.68
2	1.80	1.80
3	1.97	2.33
4	1.70	1.74
5	1.35	1.31
6	1.94	1.80
7	1.67	1.67
8	2.36	2.38
9	1.49	1.42
10	1.48	1.42
11	1.33	1.38
12 (official potash sample for 1900)	5.40	5.42 5.44 5.45

Following is the method employed:

Ignite 10 grams of sample with sulphuric acid, and after cooling add 4 or 5 cc sulphuric acid (1–1) and gently warm the dish. The sample may now be easily removed from the dish, and the small amount of free sulphuric acid interferes in no way with the following stages of the process.

Boil the ignited sample with about 350 cc of water for thirty minutes, and while hot add milk of lime till alkaline. Precipitation of all the interfering salts is complete. Cool, make up to 500 cc, take an aliquot portion, acidify with hydrochloric acid, and evaporate direct with platinum solution. Wash with alcohol and ammonium-chlorid wash water, as in the Lindo-Gladding method. Little more time is required for the washing than in the Lindo-Gladding method.

It was found that rubbing the residue quite vigorously with a rubber pestle caused low results, presumably from the passing of finely divided potassium platinic chlorid through the filter.

If a somewhat larger amount of ammonium-chlorid wash water be used than in the Lindo-Gladding process, gentle rubbing with the finger or with a rubber-tipped glass rod suffices to divide the residue finely enough for the wash water to completely dissolve the salts of lime present.

RECOMMENDATIONS.

The referee would suggest:

(1) Investigations into the causes of the low results obtained by the official method.

(2) That the "milk-of-lime" method be submitted for investigation the ensuing year.

Mr. Ross. With regard to the "milk-of-lime" method, to which considerable attention has been given by the referee, I would say that this process suggested itself to me shortly after the preparation of my report on potash in 1899, and immediately a series of tests was instituted in the Alabama State laboratory to determine the practicability of this modification. This process is, of course, only applicable to mixtures of acid phosphate and potash salts, or to fertilizers containing organic materials, in which case saturation of the material with sulphuric acid and incineration are resorted to before effecting the solution of the potash.

In the present official method for determining total potash in organic mixtures, the material is incinerated after treatment with sulphuric acid, and at a subsequent stage of the process evaporation and ignition must be again resorted to in order to remove the ammonia and ammonia salts which have been employed as precipitants. In the "milk-of-lime" process this second ignition is avoided, precipitation of phosphates, lime, alumina, etc., being effected by means of the milk of lime. Mr. Hare took this matter up even before his appointment as referee, and has performed a large amount of laborious, careful, and painstaking work in the testing of this method.

Mr. Wiley. I have a communication from Mr. Peters on this subject, which I took the liberty of opening, and I would like to submit this for publication. I do not think it is necessary to read it, although there are only two pages.

Lexington, Ky., *November 9, 1901.*

My Dear Sir: I have recently had occasion to try the determination of potassium oxid in a vegetable substance by the method of charring with sulphuric acid and burning, as described on page 22 (2 b), Bulletin 46, revised edition. The result was that less potash was obtained in this way than by boiling out with water in the manner prescribed for mixed fertilizers without previous burning. This recalls former experiences in the same line, which I think I have mentioned to you in a former letter, and makes me think that we might do well to drop the paragraph above cited from our methods, leaving determinations of total potash in such materials to be made by the methods described for the ashes of plants, and using only the method of boiling out with water for fertilizers. The old experiments referred to above were made upon tobacco stems and the results were published in the Eighth Annual Report of the Kentucky Station, 1895, page xxiii, and I have had the same experience since then. The following are the figures:

Tobacco stems, No. 2935—Air-dried sample.

	Per cent.
K_2O by boiling 10 grms. with 300 cc water one-half hour (Bull. 46, rev. ed., p. 21)	9.32
K_2O by charring with H_2SO_4, treatment with HCl, etc. (Bull. 46, rev. ed., p. 22)	9.08
K_2O by incineration and analysis of the ash	9.18

The trouble seems to me to be in not getting the mass fully dissolved by treating with hydrochloric acid and boiling one-half hour with 300 cc of water. After incinerating with sulphuric acid the mass obtained is hard and lumpy and does not readily dissolve. I believe this is a weak point in our methods and should be looked after, though it is not of very great importance, since the method is seldom needed.

Very respectfully,

ALFRED M. PETER.

Mr. C. L. HARE,
Referee on Potash, Association of Official Agricultural Chemists,
Care of Dr. H. W. Wiley, Washington, D. C.

The PRESIDENT. The report is before you for discussion.

Mr. MAY. I would like to say that I am writing a report of this meeting for the Experiment Station Record, and will ask the referees to leave their papers with Dr. Wiley and not take them home, as some have done in the past.

The PRESIDENT. I hope the various referees will adhere to the request made. If there are no remarks, I will say that opportunity will be given for the discussion of any of the reports of the referees at the time recommendations are submitted by the committee. We will proceed next to the report on phosphoric acid.

Mr. WILEY. I have a report sent by Mr. Miller, of Florida, who is unable to be present. I am suffering a little with a cold and I would ask someone to read this.

The report was read by Mr. Runyan, as follows:

REPORT ON PHOSPHORIC ACID.

By H. K. MILLER, *Referee.*

An examination of the work done the past four years in connection with the estimation of phosphoric acid by this association shows that much has been accomplished. The volumetric method has received careful study and has now passed beyond the experimental stage, and seems to give more reliable results in the hands of those experienced in its use than the gravimetric method. In view of this the referee for this year decided it would be well to pay greater attention to some other phase of the phosphoric acid work. The modification of the volumetric method as proposed by the referee of last year yielded such good results that it was deemed well to incorporate it in the instructions for this year with little change.

The usual letter was sent out to those thought to be interested, requesting cooperation in the work on phosphoric acid for 1901. Some fifty replies were received, and among these fifteen expressed a willingness to assist in the work and requested a set of samples. Samples were prepared and sent out with the following letter of instructions:

INSTRUCTIONS FOR THE WORK ON PHOSPHORIC ACID FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1901.

Three samples have been prepared for the phosphoric acid work this year.

No. 1 is a basic slag.

No. 2 is ground phosphate.

No. 3 is a mixture of salts containing phosphoric acid, alumina, and iron.

All of No. 3 is to be placed in a liter flask, a little nitric acid added, and made up to mark with water. Fifty cubic centimeters of the solution will correspond to 0.5 gram of substance.

TOTAL PHOSPHORIC ACID.

Determine total phosphoric acid in the three samples by the following methods:

(1) Official gravimetric method (Bulletin 46, revised edition, Division of Chemistry, U. S. Department of Agriculture, p. 12).

(2) Volumetric method: Make solution of the phosphate according to one of the official methods. Take an aliquot portion containing not more than 30 mg P_2O_5, add ammonium hydrate in excess, then nitric acid in slight excess, then an excess of the official molybdate solution. Place in a water bath at 45° to 50° C. for one-half hour with occasional stirring, filter, and wash with water until 25 cc of the filtrate fails to neutralize one drop of the standard potassium hydrate solution. Transfer filter and contents to the beaker. Add about 30 cc of water and sufficient standard alkali to dissolve the yellow precipitate. Titrate the excess of alkali with standard acid, using phenolphtalein as indicator. (See Bulletin 46, Division of Chemistry, U. S. Department of Agriculture, p. 13.)

CITRATE SOLUBLE PHOSPHORIC ACID.

Determine available phosphoric acid in the sample of basic slag (No. 1) as follows:

Place 3 grams of the slag in a 300 cc flask, add 200 cc of 1 per cent solution of citric acid, place in a water bath, and maintain at 50° C. for one hour. Shake vigorously five times during the digestion at intervals of about ten minutes, cool under faucet, make up to mark with water, filter out 50 cc; to this add ammonium hydrate until alkaline, then nitric acid until slightly acid, and about 10 grams of ammonium nitrate. Place in a water bath at 75° C. Add 75 cc molybdate solution, let stand thirty minutes with frequent stirring, cool, filter, wash with cold water, and proceed as under total phosphoric acid. (Bulletin 46, Division of Chemistry, U. S. Department of Agriculture, p. 12.)

IRON AND ALUMINA.

Determine iron and alumina in the three samples by the following methods:

(1) *Acetate method.*

Take 50 cc of the solution corresponding to 0.5 gram of substance; add ammonium hydrate until slightly alkaline. Add hydrochloric acid drop by drop until the precipitate just dissolves, then 2 or 3 drops more, heat to boiling, and add an excess of ammonium oxalate. Let stand until cold, filter, and wash with hot water. Treat the filtrate with ammonium hydrate drop by drop until a precipitate begins to form and then add 25 cc of a 25 per cent ammonium acetate solution. Heat to boiling, filter, and wash with a 5 per cent ammonium nitrate solution. Dissolve the residue in nitric acid, add a few drops of sodium phosphate and then ammonium hydrate until a precipitate begins to form. Treat with an excess of ammonium acetate, heat to boiling, filter, wash with ammonium nitrate, dry, ignite, and weigh as iron and aluminum phosphates.

(2) *Molybdate method.*

(*a*) To 50 cc of the solution, corresponding to one-half gram of substance, add ammonia until alkaline, make slightly acid with nitric acid, heat to 50° C, and add slowly an excess of molybdate solution, stirring constantly. Let stand until cold, filter, and wash with cold water.

(*b*) Treat the filtrate obtained under (*a*) with ammonium hydrate until nearly alkaline, add an excess of ammonium sulphid, heat to 80° C, let stand three hours, filter rapidly under pressure, and wash five or six times with water containing ammonium sulphid. Dissolve the residue in dilute nitric acid, boil a few minutes, add ammonia until slightly alkaline, and determine the iron and alumina as usual.

(*c*) Proceed as under (*a*) and to the filtrate add ammonium hydrate in slight excess. Heat to 50° C. and let stand for thirty minutes, filter, and wash with hot water. Dissolve the residue in hydrochloric acid, add ammonium hydrate until alkaline, charge the solution with excess of hydrogen sulphid, and add dilute hydrochloric acid until decidedly acid. Let stand a few minutes, filter, and wash with 1 per cent hydrochloric acid, then add a few drops of nitric acid to filtrate (which should be clear), boil to render iron in ferric condition, and determine iron and alumina in the usual manner.

In case it is found impracticable to do all the work indicated the referee would be glad to have at least the determination of iron and alumina made in Nos. 2 and 3 by the methods given. Please report results thirty days previous to the next meeting of the association.

H. K. Miller, *Referee.*
C. H. Jones, *Associate Referee.*

Results were obtained from five laboratories, including the work of eight analysts. These results appear below in Tables I and II. Before examining the results it may be well to explain briefly the points which the referee had in mind when preparing the instructions. At the last meeting of this association a resolution was passed requesting the referee for 1901 to make a study of basic slag with a view to finding a suitable method for estimating its commercial value. Accordingly, a sample of basic slag was sent out with the instructions for a trial method for approximating its available phosphoric acid. Aside from this, and a further test of the volumetric method as proposed by Runyan, it was thought advisable to make an effort to find a satisfactory method for estimating iron and alumina in the presence of phosphoric acid. Samples Nos. 2 and 3 were prepared with special view to this line of work. While many believe a satisfactory solution of this problem is to be found in some modification of the acetate method, others have been unable to find any encouragement through its use. The methods sent out by the referee are simply modifications of methods formerly proposed. Results obtained by two other modifications of the acetate method were reported and are included in the results. Table I contains the results obtained on total phosphoric acid and the results on the phosphoric acid in the Thomas slag soluble in a 1 per cent citric acid solution.

TABLE I.—*Results on phosphoric acid.*

Analyst.	Volumetric method.			Gravimetric method.			Citric acid.
	No. 1.	No. 2.	No. 3.	No. 1.	No. 2.	No. 3.	No. 1.
F. B. Carpenter, Richmond, Va	....	....	....	18.81	35.98	20.33	....
G. A. Hanvey, Richmond, Va	18.55	35.95	20.33	18.66	35.90	20.37	....
E. G. Runyan, Washington, D. C	18.03	35.07	19.90	17.84	35.10	19.94	6.08
J. G. Heavener, Richmond, Va	....	....	....	....	[b]35.60	....	....
	....	35.73	....	[b]18.39	[b]35.60	....	....
	[a]18.46	35.61	....	[b]18.43	[b]35.60	....	....
	[a]18.39	....	19.91	[b]18.39	35.60	....	....
	....	35.76	19.91	....	35.55	....	....
	[a]18.47	35.77	....	[c]18.33	....	....	....
	[a]18.46	....	....	[c]18.33	[a]35.60	....	....
	....	....	....	....	[a]35.58	....	....
B. F. Robertson, Clemson College, S. C.	18.47	35.40	20.01	18.65	35.67	20.13	....
	....	....	....	....	....	20.32	6.75
C. C. McDonnell, Clemson College, S. C.	18.32	35.58	20.50	18.62	35.64	20.10	....
	....	....	....	....	....	20.44	6.82
H. K. Miller, Lake City, Fla	....	35.80	....	....	....	....	....
	18.20	35.70	....	18.26	35.63	....	....
	18.40	35.60	....	18.34	35.65	....	....
A. W. Blair, Lake City, Fla	18.85	36.05	....	....	....	....	....
	19.05	35.90	....	....	....	....	....
	19.02	36.07	20.57	....	....	....	....
	19.07	36.07	20.55	18.85	35.69	20.30	6.79
	18.68	35.70	20.62	18.75	35.74	20.29	6.73
	18.80	36.00	20.50	....	....	20.44	6.78
	18.82	35.77	20.55	....	....	20.58	6.79
	18.85	35.72	20.55	....	....	....	....

[a] Precipitated at room temperature.
[b] Dissolved in sulphuric acid.
[c] Silica removed.

Table II contains the results obtained on the iron and alumina in samples Nos. 2 and 3.

TABLE II.—*Results on iron and alumina.*

Analyst.	Carpenter's acetate method.		Referee's acetate method.		Molybdate method B.		Molybdate method C.	
	No. 2.	No. 3.	No. 2.	No. 3.	No. 2.	No. 3.	No. 2.	No. 3.
E. G. Runyan, Washington, D. C	[a]3.70	[a]4.38	2.80	2.58	7.20	7.85	2.50	4.33
F. B. Carpenter, Richmond, Va	3.02	4.63						
G. A. Hanvey, Richmond, Va	2.95	4.50						
S. H. Sheib, Richmond, Va	2.83	4.53						
C. C. McDonnell, Clemson College, S. C			2.30	4.20			2.50	4.11
B. F. Robertson, Clemson College, S. C			2.98	4.06	2.77	3.51		
A. W. Blair, Lake City, Fla			2.64	2.22				
		4.04	2.38	3.69				
		3.95	2.44	2.84				
				1.48				
H. K. Miller, Lake City, Fla					6.68	6.74		
					6.28	6.82		
Actual		4.51		4.51		4.51		4.51

[a] Acetate method (Bull. 62, Div. Chem., p. 35).

NOTES BY THE ANALYSTS.

F. B. Carpenter.—In the volumetric method we used a temperature of from 60° to 65° C., allowing the solution with the precipitate to remain in the bath about twelve minutes. All the methods for iron and alumina were tried, but none proved satisfactory. In the case of the basic slag the results do not agree as well as with the other samples, and we had the same experience last year with material of this kind. I think that the trouble probably lies in the fact that there is a large amount of soluble silica present, which, in case of the gravimetric method, may be precipitated to some extent along with the magnesia mixture.

E. G. Runyan.—In some cases the determinations were made in sextuplicate and in no case less than duplicate. Judging from the agreement of the results the figures on phosphoric acid are fairly satisfactory. The results on iron and alumina are rather wild, but I am not willing to admit that this is entirely the fault of the analyst. I believe the methods proposed by the referee are faulty, as they embrace steps which McElroy, Krug, and others in this laboratory have shown to yield unreliable results. With each method the determinations were made in duplicate and the averages given in the table represent a fair trial of the proposed method.

J. G. Heavener.—The white precipitate of sample No. 1 still had traces of iron after two precipitations.

A. W. Blair.—It was found impossible to obtain a perfect solution of No. 1 by the usual methods employed. It was necessary to dissolve on the water bath in order to prevent the formation of a gelatinous precipitate, which always interfered with a perfect solution, inasmuch as it adhered to the bottom of the flask when heated on a hot plate.

B. F. Robertson.—The ferric oxid from the ammonium sulphid precipitate contained some manganese, which was separated by dissolving in hydrochloric acid and precipitating the ferric oxid with ammonium acetate.

C. C. McDonnell.—The iron and alumina precipitate from No. 1 (molybdate method) contains some manganese, which accounts for the alumina being so high. In this determination also there was a loss of iron due to the fact that on acidifying the sulphids with hydrochloric acid some sulphid of iron was left undissolved. The results on phosphoric acid are averages of several determinations. The results with the volumetric method show wide variations in some cases.

DISCUSSION OF RESULTS ON PHOSPHORIC ACID.

On examining the results by the volumetric method on sample No. 1 it will be seen that rather wide variations occur in one or two cases. The results are too low in a few cases, and I think it can be safely assumed that this is due to imperfect solution and not to the volumetric method proper. The results reported by one analyst seem rather high. Altogether, with these exceptions, the results given are quite satisfactory. I believe the same trouble has been experienced with sample No. 2. A few of the results are somewhat low; only one, however, is too low to be included in the averages. I am inclined to believe the high results represent more nearly the actual phosphoric acid present in samples Nos. 1 and 2 than the average. It has been my experience that it frequently occurs in dissolving certain phosphates and Thomas slag that solution is not complete even after prolonged digestion with fresh additions of acid. It has been shown, too, that during the digestion it sometimes occurs that the dissolved phosphate becomes caked on the side of the flask above the surface of the acid and is thus rendered insoluble. In the case of Thomas slag I have been unable to get a perfect solution when strong acids were poured on the material and the flasks placed on the hot plate. The variations obtained on No. 3 by the volumetric method are only slight, except in one or two cases. Passing to the gravimetric method we find in the case of No. 1 that the variations are greater than in the case of the volumetric method. With No. 2 by this method it will be seen that the greatest variations occur. The result on No. 3 by the gravimetric method are in one or two cases a little wild, but on the whole are fairly satisfactory. From the results obtained last year and those of this year, and from my experience with the modification of the volumetric method as proposed by Runyan, I think it is capable of giving highly satisfactory results and of being expeditiously handled. The phosphoric acid in sample No. 1, soluble in a 1 per cent citric acid solution, is reported by four analysts with fairly concordant results.

DISCUSSION OF RESULTS ON IRON AND ALUMINA.

As usual few results were reported on iron and alumina, and, as is generally the case, quite varying results have been obtained. The results on No. 1 have not been included in the table, inasmuch as the manganese present interfered with the determination of iron and alumina and those reported were exceedingly wild, varying from 3 to 20 per cent. The results obtained on Nos. 2 and 3 by the acetate method as modified by Carpenter are quite satisfactory compared with previous reports. It will be seen from the few results reported that the referee's proposed acetate modification does not yield good results. This is true also of the molybdate method B, but the molybdate method C yielded fairly concordant results. It is not possible to draw any conclusion from the results obtained on the iron and alumina, inasmuch as the data is too meager.

RECOMMENDATIONS.

It would evidently be unwise to make any recommendations based on the results which have been reported on these samples. It is quite evident, however, that one question should receive some action by this association, and that is a method applicable to Thomas slag. While I am not prepared to make a direct recommendation in regard to this, I think it would be well for the committee to consider the advisability of adopting some form of the citric acid method. The Wagner method, I think, comes in for the most favorable consideration. It must be borne in mind that "available phosphoric acid" is an unwise term, and I think it would be well to have in view the idea of valuing fertilizers according to the constituents found therein. This applies especially to phosphoric acid, and inasmuch as we have several materials containing phosphorus, but in different combinations, which are useful as fertilizers, it is not probable that a single method will be found applicable to all these. The only solution apparent at this time is to recognize this difference and apply special methods.

The President. Are there any other papers to be presented in relation to phosphoric acid—any remarks or discussion?

Mr. Huston. Are there any specific recommendations in this report?

The President (to Mr. Wiley). What is the particular point with reference to any recommendations of a general kind in the report?

Mr. Wiley (reading from the recommendations of the referee for the information of Mr. Huston). "While I am not prepared to make any direct recommendations," etc.

Mr. Huston. Although there are no direct recommendations, the report is to go to the committee, as there may be some points in it that we might take up. We tried this course at least ten years ago and I do not see the use of trying the same thing over and over again. If we take it up at all we must proceed on an entirely different basis. I do not think it difficult to determine the amount of iron in these samples. It can be done without trouble. I have had very excellent results on iron, and there was no difficulty in handling the alumina, but I think we ought to proceed on an entirely different basis. I appreciate the fact that a majority of the former members of this association were anxious to have iron and alumina included, but even if they were trying to follow that method I think we ought to take a new start and see if we can not get hold of some method, even if it is a long one, which will finally determine these points; but the method will have to be wholly different from any we have been using so far. I think it can be done, but I do not think we are on the right track.

The President. Is the committee which was appointed to consider and report upon changing the number of committees on recommendations of referees ready to report? If so, the report will be received at this time.

REPORT OF COMMITTEE ON CHANGING NUMBER OF COMMITTEES ON RECOMMENDATIONS OF REFEREES.

The committee to whom was referred for consideration and report the question of the number of committees on recommendations desirable for our present work would recommend that, instead of one committee, as heretofore, three committees of five members each be appointed, with the following assignment of subjects to each:

A. Phosphoric acid, potash, soils, ash, and insecticides.
B. Dairy products, foods and feeding stuffs, sugar, tannin.
C. Liquors and detection of food adulteration.

William Frear,
H. J. Wheeler,
B. W. Kilgore,
Committee.

Mr. May. I move the adoption of the report.

The President. A motion has been made and seconded that the report of the committee as read be adopted. Discussion is in order. The report is adopted.

The report of the referee on soils is next in order.

Mr. Wiley. Mr. Jaffa unfortunately can not be here, but he has sent his report, which is ready for presentation. This report begins with a statement of the work to be done.

REPORT ON SOILS.

By M. E. Jaffa, *Referee.*

The work of the referee on soils was confined this year to the further investigation of methods for obtaining the available or assimilable plant food. The analytical data here reported are gained from five soils—two from California and three from different sections of the Eastern States—the object being to gather data from as many different kinds of soils as possible. Credit is due Mr. C. A. Triebel for material assistance in the chemical work here reported.

The circular letter given below was sent to the different experiment station chemists asking for cooperation in the work for the year. Thirteen favorable replies were received, but analytical data have been received from only three stations,[a] viz, Oregon, Ohio, and Virginia. It is to be exceedingly regretted that more results are not at hand for comparison.

March 23, 1901.

Dear Sir: The referee on soils of the Association of Official Agricultural Chemists this year will continue the study of methods for the determination of available mineral plant food; also the question of humus and humus nitrogen. We would like to have your cooperation in the work. Please notify the referee if you will undertake it.

M. E. Jaffa, *Referee.*
F. P. Veitch, *Associate Referee.*

The working directions which were sent to the respondents to the circular letter are as follows:

Directions for Work on A. O. A. C. Soil Samples for 1901.

I.—THE DETERMINATION OF MOISTURE.

Use the official method described in Bulletin 46, page 71, Division of Chemistry, U. S. Department of Agriculture. Calculate all results to the water-free basis.

II.—THE DETERMINATION OF AVAILABLE POTASH SOLUBLE IN N/5 HCl.

(*a*) *Preliminary treatment.*—Digest 20 grams of soil with 200 cc of N/5 HCl, in a water bath, at 40° C., for five hours, shaking every half hour. Titrate 20 cc of the clear filtrate against a standard KOH solution, using phenolphthalein for the indicator. From the data thus secured calculate the amount of HCl necessary to be added so that the solution will be N/5 after allowing for the acid neutralized.

(*b*) *The determination.*—Two thousand to 3,000 cc of acid, corrected for neutralization, as directed under (*a*), are heated in a stopped receptacle to 40° C. Ten grams of soil for every 100 cc of acid are now added and the digestion continued at this temperature for five hours, shaking thoroughly every half hour. The contents are then shaken and filtered through a dry, ribbed filter of two thicknesses of paper, refiltering the first portion of the filtrate if cloudy. Take an aliquot portion of the filtrate, evaporate to a small volume in a porcelain dish, transfer to a platinum dish, add a little sulphuric acid, and determine the potash by the Lindo-Gladding method.

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[a] Subsequent to the reading of the report, data were received from two additional sources, i. e., Kentucky Agricultural Experiment Station and Division of Soils, U. S. Department of Agriculture, Washington, D. C.

III.—THE DETERMINATION OF AVAILABLE POTASH AND PHOSPHORIC ACID SOLUBLE IN 2 PER CENT CITRIC ACID SOLUTION.

(*a*) *Preliminary treatment.*—Determine, in separate portions of the soils, the amount of carbonic acid. From the data thus obtained calculate the amount of citric acid necessary to be added so that the solution will be 2 per cent after allowing for the carbonic acid expelled.

(*b*) *The determination.*—Digest 50 grams of soil with 300 cc of 2 per cent citric acid solution, with frequent shakings, for twenty-four hours. Filter through two thicknesses of paper. Evaporate filtrate to a small volume in a porcelain dish, transfer to a platinum dish, evaporate to dryness, carefully ignite the residue, digest with hydrochloric acid and water (sol. A).

Take two aliquot portions of the sol. A. Use one portion for determining phosphoric acid according to one of the official methods. Evaporate the other portion to a small volume, if necessary, in a porcelain dish, transfer to a platinum dish, add a little sulphuric acid, and determine the potash by the Lindo-Gladding method.

IV.—HUMUS AND HUMUS NITROGEN.

Determine humus and humus nitrogen in accordance with directions given on page 76, Bulletin 46, revised edition, Division of Chemistry. In addition thereto transfer an aliquot of the ammoniacal humus solution, prepared as directed, to a flask suitable for Kjeldahl digestion, evaporate to dryness on water bath and then expose to temperature of 100° C. in order to drive off the *free* ammonia. Treat the residue thus obtained with MgO, and thus determine the amount of *combined* ammonia.

Transfer another aliquot of the ammoniacal humus solution to a Kjeldahl flask, evaporate as above, and determine the total nitrogen in the residue by the Kjeldahl method. The question is, does the nitrogen obtained by treatment with NaHO solution bear any relation to the amount of nitrogen contained in the residue from the ammonia treatment, after the combined ammonia (expelled by MgO) has been deducted therefrom?

DESCRIPTION OF THE SOILS.

No. 1.—"Adobe soil" from the moist lands bordering on Chino Creek, Los Angeles County, Cal., taken to depth of 12 inches. A mouse-colored, moderately clayey loam, somewhat silty; the dry lumps crush quite readily between the fingers and show little or no grit. With acid there is effervescence on wetting, the color deepens considerably, and when kneaded the soil becomes quite adhesive, showing that it must not be tilled while wet. Its natural vegetation is a dense, tall growth of grass, with some spots bearing the yerba mansa (*Anemopsis californica*) and sunflower. The complete analyses are as follows:

	Per cent.
Coarse material above 0.5 mm in diameter	10.00
Fine earth	90.00
Total	100.00

Chemical analysis of fine earth.

		Per cent.
Insoluble matter	62.62	
Soluble silica	8.30	
		70.92
Potash (K_2O)		.95
Soda (Na_2O)		.50
Lime (CaO)		5.07
Magnesia (MgO)		.84
Trimanganese tetroxid (Mn_3O_4)		.06
Ferric oxid (Fe_2O_3)		6.43
Alumina (Al_2O_3)		4.88
Phosphoric acid (P_2O_5)		.21
Sulphuric acid (SO_3)		.06
Carbonic acid (CO_2)		3.76
Water and organic matter		6.02
Total		99.70

Mechanical analysis.[a]

	Per cen
Colloid clay, per cent	13.44
Silt:	
0.25 mm, hydraulic value	20.65
0.25 mm, hydraulic value	1.37
0.50 mm, hydraulic value	4.91
1 mm, hydraulic value	5.95
2 mm, hydraulic value	13.46
4 mm, hydraulic value	10.36
8 mm, hydraulic value	9.25
16 mm, hydraulic value	12.01
32 mm, hydraulic value	2.75
64 mm, hydraulic value	1.66
	95.81
Water capacity	60.20

No. 2.—Orange-tinted loam from Arlington Heights, San Bernardino County, Cal. The soils of this tract are of great depth; in the breaks profiles of as much as 30 feet of sensibly uniform red loam may be seen, and it will be noted by reference to the following analyses that it is of almost uniform composition, chemically, to the depth of 10 feet.

The roots of wild shrubs are found within it to depths of from 6 to 8 feet, showing it to be as easily penetrable by roots as it is by water, its red tint alone being proof of its perfect drainage.[b] Vegetation: Cactus, baeria, croton, and greasewood.

Chemical analyses of soil and subsoil of Arlington Heights.[a]

	Soil No. 2.	Subsoil No. 2.
Depth feet	1	10
	Per cent.	*Per cent.*
Coarse material, > 0.5 mm	20.00	23.00
Fine earth	80.00	77.00
	100.00	100.00
Insoluble matter and soluble silica	[b] 84.61	[c] 83.51
Potash (K_2O)	.87	.93
Soda (Na_2O)	.29	.36
Lime (CaO)	1.57	1.73
Magnesia (MgO)	1.33	1.58
Trimanganese tetroxid (Mn_3O_4)	.04	.04
Ferric oxid (Fe_2O_3)	4.20	5.34
Alumina (Al_2O_3)	5.30	5.08
Phosphoric acid (P_2O_5)	.14	.16
Sulphuric acid (SO_3)		
Water and organic matter	1.60	1.44
	99.85	100.17
Hygroscopic moisture absorbed at 15° C	1.77	2.16

[a] Cal. Sta. Report, 1890, p. 41.
[b] Insoluble matter, 76.41; soluble silica, 8.20.
[c] Insoluble matter, 75.44; soluble silica, 8.07.

Soils Nos. 1 and 2 were selected for distribution because they are both virgin, productive soils, showing high percentages of potash and phosphoric acid, but decidedly different in character and humus content, No. 1 being rich and No. 2 poor in this respect.

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[a] Dr. R. H. Loughridge, analyst.

[b] Cal. Sta. Report, 1890, p. 39.

Soils Nos. 3, 4, and 5 were kindly furnished by Mr. B. L. Hartwell, of the Rhode Island station. The field records of these soils will be found in the Proceedings of the Seventeenth Annual Convention of the Association of Official Agricultural Chemists, Bulletin 62, Division of Chemistry, United States Department of Agriculture, pages 75–77.

The percentages of fine and coarse earth, as obtained by Mr. Hartwell, in soils Nos. 3, 4, and 5 are given in the subjoined table:

	No. 3. Wapping soil.	No. 4. Lime rock soil.	No. 5. Amherst soil.
	Per cent.	*Per cent.*	*Per cent.*
Coarse earth, >0.5 mm	16.50	13.90	2.40
Fine earth	83.50	86.10	97.60
Total	100.00	100.00	100.00

The fine earth in each case was distributed, as it is this part of the soil that forms the storehouse of available plant food.

POTASH.

It was desired this year to make further studies of the citric acid and N/5 hydrochloric acid methods for determining the amount of available potash and phosphoric acid contained in soils, more particularly as in the case of Nos. 3, 4, and 5 we had the data[a] obtained by Mr. Hartwell in his past experiments in those soils for reference and comparison.

Unfortunately we have far too few results to enable us to draw any conclusions whatever.

It appeared to the referee that more extended work on the citric acid method was called for. Citric acid certainly approaches more nearly to the root acid than any of the mineral acids, and also cold digestion is closer in touch with the processes of nature than is a temperature of 40° C.

The citric acid method is the one in use at the California station, and the results agree satisfactorily with actual practice. Dr. Dyer recommends a 1 per cent solution of citric acid; but, owing to the generally high content of lime in California soils and to the fact that the acidity of citrus roots is quite often over 1 per cent, it was deemed advisable to use a 2 per cent solution, after allowing for the carbonate of lime which the soil might contain.

TABLE I.—*Potash (K_2O) soluble in N/5 HCl.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. W. Ames, Ohio	....	0.0299	0.0110	0.0155	0.0175
Ellett and Gibboney, Virginia	0.1231	.0284	.0150	.0119	.0124
Major Edwards, Oregon	.0830	.0610	.0280	.0310	.0360
M. E. Jaffa, California	.1149	.0298	.0205	.0174	.0201
F. P. Veitch, Washington, D. C.[b]	.0938	.0139	.0242	.0311	.0155

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[a] U. S. Dept. of Agr., Bureau of Chem. Bul. 62, p. 73.

[b] These data were received too late to be included in the discussion of results and general averages, but are added to the table.

DISCUSSION OF RESULTS.

An examination of Table I shows soil No. 1 to be very rich in available potash, and the results obtained by the Virginia and California stations agree quite closely; similar conditions obtain for soil No. 2, if we leave out of consideration the data obtained by the Oregon station. The figures given for Nos. 3, 4, and 5 are not very satisfactory.

The maxima per cents are quoted by the Oregon station in each case, being, respectively, 0.028, 0.031, and 0.036. The minimum for the soil No. 3 is 0.011 per cent, to the credit of the Ohio station, and the minima per cents for Nos. 4 and 5, 0.0119 and 0.0124, respectively, are given by the Virginia station. The average per cents of all the determinations are as follows:

No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
0.107	0.0373	0.0186	0.0189	0.0215

If we omit the figure for No. 2, obtained by the Oregon station, the average will then be 0.0294, which would seem to be more correct for the average than the previous one.

It is a matter of regret to the referee that he can not present more data on the citric acid method than those which are given in the following table:

TABLE II.—*Potash* (K_2O) *soluble in 2 per cent citric acid.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Major Edwards, Oregon	0.083	....	0.014	0.018	0.023
M. E. Jaffa, California	.056	0.017	.016	.014	.017

The experience of the California station has shown that the results yielded by this method are always less than the corresponding ones reported for the hydrochloric acid treatment. The figures given in Table II are in accordance with our other experiments in this direction.

The above data are far too meager to allow of summarizing in any definite way. But if a tentative conclusion could be ventured we might say, in accordance with our experience in California and the results as shown by the careful and able experiment[a] of Mr. Hartwell, that any soil showing 0.02 per cent of K_2O by either of the above methods is not in pressing need of potash fertilization. More work along this line is urgently called for. The citric acid method certainly should not be discarded without proper and careful investigation.

PHOSPHORIC ACID.

In Table III are given the results obtained for phosphoric acid by the citric acid digestion.

TABLE III.—*Phosphoric acid soluble in 2 per cent citric acid.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
Major Edwards, Oregon	0.012	0.019	0.025	0.018	0.023
M. E. Jaffa, California	.031	.027	.033	.016	.024
F. P. Veitch, Washington, D. C. [b]	.181	.055	.133	.044	.021

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[a] U. S. Dept. Agr., Bureau of Chem Bul. 62, p. 73.

[b] Data received too late to be included in discussion of results or in the general averages, though included in the table.

Again, we find that there has not been sufficient cooperation to warrant our forming any conclusions; and in view of the importance of adopting some method for determining the available plant food in soils, one which will yield concordant results in the hands of expert chemists, and also because so little cooperation is afforded the referee on soils each year, I would offer the following suggestion: That a large committee of chemists be appointed, with the referee on soils as chairman, to investigate and report upon some method for determining the available plant food in soils in place of continuing the practice now in vogue.

HUMUS AND HUMUS NITROGEN.

The object of the work along this line was to ascertain, if possible, the relation, if any, which exists between the nitrogen extracted by ammonia and that contained in the sodium hydrate or potassium hydrate leachings; but, as evidenced by the tables below, far too few data have been gathered to enable us to arrive at any conclusions.

In Table IV are given the results obtained for humus:

TABLE IV.—*Humus in soils.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Ellett and Gibboney, Blacksburg, Va	2.52	0.81	4.24	2.12	3.80
Major Edwards, Corvallis, Oreg	2.60	.89	2.45	2.02	2.04
M. E. Jaffa, Berkeley, Cal	2.14	.42	2.44	2.03	2.04

The discrepancies here shown are far from satisfactory, and the same may be said with respect to Table V, showing percentages of nitrogen in residue from ammonia leachings:

TABLE V.—*Total nitrogen in residue.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Ellett and Gibboney	0.437	0.203	0.448	0.455	0.466
M. E. Jaffa	.228	.084	.232	.171	.202

The above differences are somewhat difficult to account for in view of the fact that the figures for the nitrogen as combined ammonia in the same residue do not show similar discrepancies. This is seen by a glance at the following statement:

TABLE VI.—*Nitrogen,*[a] *as combined ammonia in residue from ammonia leachings.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Ellett and Gibboney	0.053	0.031	0.089	0.052	0.159
M. E. Jaffa	.054	.026	.073	.067	.073

[a] Determined by treatment with magnesium oxid.

Subtracting the respective results contained in Tables V and VI, and arranging, we have the following:

Table VII.—*The "organic" nitrogen in the residue from ammonia leachings.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Ellett and Gibboney	0.384	0.172	0.359	0.403	0.307
M. E. Jaffa	.174	.058	.159	.104	.129

Comparing the figures in Table VII with the data reported for the humus nitrogen dissolved by the sodium hydrate solution, we have:

Table VIII.—*Comparison of humus nitrogen extracted by ammonia and sodium hydrate solution.*

Analyst.	No. 1.		No. 2.		No. 3.		No. 4.		No. 5.	
	Ammonia.	Sodium hydrate.	Ammonia.	Sodium hydrate.	Ammonia.	Sodium hydrate.	Ammonia.	Sodium hydrate.	Ammonia.	Sodium hydrate.
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Ellett and Gibboney	0.384	0.173	0.172	0.043	0.359	0.065	0.403	0.096	0.307	0.106
Major Edwards	....	.141	....	.061	....	.147	....	.145	....	.078
M. E. Jaffa	.174	.171	.058	.059	.159	.160	.104	.117	.129	.132
Average	....	.162	....	.054	....	.124	....	.119	....	.105

The above is not a very satisfactory showing. While there is agreement between some of the results reported for the nitrogen by sodium hydrate, the percentages of this element extracted by the ammonia solution are far from being concordant.

Again, the data presented by the California station would indicate that there is a relation between the nitrogen dissolved by sodium hydrate and that by ammonia, and, further, that after deducting the amount of nitrogen in the form of combined ammonia, the percentages of organic nitrogen by treatment with sodium hydrate and ammonia, respectively, are practically identical in Nos. 1, 2, 3, and 5. The presentation by the Virginia station, however, shows no such relation, the amount of organic nitrogen by the ammonia treatment far exceeding that by sodium hydrate.

The determinations of humus nitrogen by sodium-hydrate digestion were checked by similar treatment with 5 per cent potassium hydrate solution, with the result as shown in Table IX, which also contains the complete data obtained by the California station for humus and humus nitrogen.

Table IX.—*Humus and humus nitrogen.*

No.	Humus.	Humus nitrogen in soil.					Humus nitrogen in humus.			
		Nitrogen as combined ammonia.	Total nitrogen in humus.	"Organic" nitrogen.	Nitrogen in KOH leachings.	Nitrogen in NaOH leachings.	Nitrogen as combined NH_3 in humus.	Organic nitrogen in humus.	Nitrogen in KOH leachings.	Nitrogen in NaOH leachings.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	2.14	0.054	0.228	0.174	0.177	0.171	2.52	8.13	8.25	8.00
2	.42	.026	.084	.058	.062	.059	6.19	13.83	14.76	14.05
3	2.44	.073	.232	.159	.162	.160	3.00	6.51	6.64	6.56
4	2.03	.067	.171	.104	.114	.117	3.30	5.12	5.62	5.76
5	2.04	.073	.202	.129	.132	.132	3.58	6.32	6.47	6.47

An examination of this table proves that the amounts of nitrogen dissolved by potassium hydrate and sodium hydrate, respectively, are identical, and also that while the percentage of humus in Nos. 3, 4, and 5 are high, that of the humus nitrogen is correspondingly low.

In conclusion, the referee would recommend that extended investigation be carried on along this line of work, but, as previously suggested, owing to the lack of sufficient cooperation, more profitable results would in all probability be obtained if the subject were left for consideration to a committee with the referee on soils as chairman.

ROTHAMSTED METHOD OF SAMPLING SOILS.

In accordance with the recommendations made at the last meeting of the association, the referee submits the following on this subject. The writer is in heartiest sympathy with Dr. Dyer in his desire to obtain a uniformity of method for the sampling of soils, and feels with him that the importance of the subject warrants continued and determined effort to accomplish that end.

The taking of the sample is one of the most vital parts of soil investigation, and we can never arrive at accurate and uniform results until the first and crucial step in the process has been thoroughly discussed and the best method suitable for all places and conditions decided upon.

The method[a] as practiced at Rothamsted is as follows:

> The following plan has been used in Rothamsted since 1856. A frame made of stout sheet iron, in shape a rectangular prism, open at top and bottom, is driven into the soil by repeated blows of a wooden rammer, till the soil has the same level inside and outside the frame. The soil inside the frame is then cut out and constitutes the sample of the first depth or surface soil. That the frame is accurately emptied is ascertained by trials with a wooden gauge of the same depth as the iron frame. If a sample of the next depth is to be taken, the soil is cleared away around the outside of the frame till the level is reduced to that of the bottom of the frame; the frame is then driven down again and the former operations are repeated.
>
> Soil sampling at Rothamsted is usually carried down to three depths, but in a good many cases it has been carried down to twelve depths. The area of the sampling frame used for the first depth is usually 144 square inches (12 by 12 inches), a smaller frame, 6 by 6 inches, being used for the succeeding depths. The depth of each frame is 9 inches. Messrs. Lawes and Gilbert are of the opinion that 6 inches would have been a better depth to adopt for the samples of surface soil, but so much work has been done with 9 inches that it would now be very inconvenient to make any alteration.
>
> The iron frame has a stout rim along its upper edge to increase its strength. The best sampling frame is made of cast steel; this form of frame needs no rim. Models both of the larger and smaller steel frames used at Rothamsted are before you, and I am instructed by Sir John Lawes to leave these models with your Department of Agriculture.
>
> When the soil sampling is carried below the first depth care must be taken when digging around the frame that each depth of soil removed is placed by itself, so that when the pit is filled in the soil may be returned to its proper position. A record must be kept of the place where the sampling was conducted, as a soil can not be accurately sampled twice in the same place.
>
> Each sample of soil is weighed as soon as it is removed from the frame, and is put into a bag by itself. When the soil reaches the laboratory it is at once broken up by hand into small pieces and laid on paper trays, which are placed on the shelves of a storeroom kept at a temperature of about 55° C. till thoroughly dry; each sample is then returned to its bag. This immediate drying of the soil at a low temperature is essential, if changes in the organic matter, and especially nitrifaction, are to be stopped. This practice dates at Rothamsted from 1877. After drying, the soil may be stored till leisure is found for further work. Each bag is then weighed. The soil is crushed and passed through a one-fourth inch sieve; the stones that do not pass through this sieve are weighed as stones. All that passes through the sieve is thoroughly mixed and a sufficient quantity is finely powdered for analysis. Mixed samples are prepared after the soil has passed through the one-fourth inch sieve, or after it has reached the stage of fine powder.

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[a] U. S. Dept. Agr., Office of Expt. Sta. Bul. No. 8, p. 39.

Dr. Dyer states:

> This method of taking soil samples, with the exception of the adoption of the definite temperature above mentioned for drying, has been, as has already been said, in use in Rothamsted since 1856, and it is interesting here to record that between 4,000 and 5,000 individual samples of the Rothamsted soils have been collected in the fashion above described.

In view of these facts it can be easily understood why this plan of sampling should not be changed for Rothamsted. But in a new country it is an entirely different question, and we ought to adopt some uniform method of sampling soils. It seems to the writer, however, that the Rothamsted method has several objections outlined below:

(1) The sampling tool, as described, is much to heavy for rapid work.

(2) When a virgin soil is sampled more than the requisite quantity of soil is collected.

(3) If a fertilized field is in question, then more samples should be gathered and the average taken. This with the Dyer method would entail a large amount of work.

(4) It is almost impossible by this method to obtain an accurate sample of the surface soil unless it should be just 8 or 9 inches in depth.

Some surface soils are 6 inches, others 12 inches in depth, and then again, as in some localities of California, the surface soil is much deeper.

Such a practice as described by Dr. Dyer would utterly fail of its purpose if applied to the larger part of the arable lands of California.

It would seem that the post-hole auger, which has been used with success in this State since its adoption by Dr. Hilgard, is much better adapted to the sampling of soils than is the implement suggested by Dr. Dyer, because—

(1) Less work is required in taking the sample.

(2) More samples can be collected in a given time.

(3) The exact point of change from surface to subsoil can be ascertained. No arbitrary depth can be, or should be, assigned for surface soil. It depends upon the depth of the humus layer; that and that only should, as a rule, constitute the surface soil. When, however, this layer is only about 3 inches deep, then the case is somewhat different and at least 6 inches should be taken.

(4) Much less bulk has to be handled.

The foregoing are suggestions offered for the consideration of the association.

It must be remembered that at the meeting three years ago a conclusive report was rendered on the question of a uniform fertilizer-control law by a committee composed of some of the most prominent members of the association. In view of the manifest necessity of a rational system of sampling of soils and of the adoption of a method for determining their available plant food, the referee suggests that a similar committee be appointed for the consideration of these subjects.

Mr. WITHERS. I have a report on soils which I would like to present to this committee.

The PRESIDENT. If the report is on soils it may be read at this time.

THE NITRIFICATION OF AMMONIUM SULPHATE AND COTTON-SEED MEAL IN DIFFERENT SOILS.

By W. A. WITHERS and G. S. FRAPS.

In Bulletin No. 176 of the North Carolina Agricultural Experiment Station, some account of which was read before this association at its meeting in 1900, the authors reported the results obtained by making determinations of the amounts of nitrates present in a soil with which different samples of various nitrogenous materials had been in contact for about three weeks, in some cases with and in others without the addition of calcium carbonate. It was found, as a result of that series of experiments, that, with the exception of ammonium sulphate, the substances which were more readily nitrified were more readily available, the availability being determined by the neutral permanganate method and also by vegetation tests with oats and Hungarian grass.[a] This order was dried blood, cotton-seed meal, dried fish, tankage, and finally bone. It was found also that the rate of nitrification was increased by dilution and by the addition of calcium carbonate. The low rate of nitrification of ammonium sulphate was confirmed by a second series of experiments. The following explanation of the low rate of nitrification of ammonium sulphate was offered by us:[b]

> The low rate of nitrification of ammonium sulphate is probably due to the presence of organisms which nitrify organic compounds in preference to ammonium salts. The presence of the ammonium sulphate may also hinder the activity of the nitrifying organisms. The acids formed may also be a hindrance when no base is present to neutralize them. All three of these causes may be in operation at the same time.

PLAN OF THE EXPERIMENT.

The object of the present experiments was to test the correctness of the above explanation. The experiments upon which we wish to report at this time were conducted upon the same general plan as our former experiments. The sample of soil in each case was sifted through a coarse sieve, and to 500 grams was added an amount of material (ammonium sulphate or cotton-seed meal) containing exactly 0.3 gram of nitrogen. The soil and its contents were thoroughly mixed, transferred to a glass precipitating jar, and placed in a dark closet. Enough water was added from time to time to bring the amount to nearly 15 per cent, the amount which it was necessary to add being determined by weighing one or more of the jars in each set and estimating the loss.

In those cases where calcium carbonate was added the amount which was used was exactly sufficient to combine with the sulphuric acid and the nitrogen of the ammonium sulphate, if the entire amount of the latter were converted to nitric acid.

At the end of three weeks the nitrates were leached out and the amount determined by the Tiemann-Schulze method. The percentages recorded were calculated after deducting the amount of nitrates formed in a blank experiment.

Tests were made for hydrogen sulphid in several cases by placing paper moistened with lead acetate over the jar containing the soil mixture and over the mouth of a flask containing the soil extract acidified and raised to boiling.

SAMPLES OF SOIL USED.

The samples used in the experiment came from localities far removed from each other. They are varied not only as to their natural character, but also in the method of fertilizing, crop grown, etc. They are as follows:

No. 1667.—Pasture soil from the North Carolina station. A light loam containing humus and not acid to litmus.

No. 1668.—Heavy clay soil from the North Carolina station. Contains no humus, possesses low fertility, and is slightly acid to litmus.

No. 1669.—Black garden soil from the Florida station. Contains much humus and is acid to litmus.

No. 1670.—From the Massachusetts Hatch station. Contains little humus and is acid to litmus. This soil is from field C, which has been used in plat experiments for some time, and since 1891 has been fertilized with sulphate of ammonia, muriate of potash, dissolved bone black, and stable manure.

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[a] Jenkins & Britton, Conn. State Station Report, 1897, p. 357.

[b] Bul. No. 176, North Carolina Station, p. 22.

No. 1674.—From the Rhode Island station. Contains humus, is slightly acid to litmus, and has been used in plat experiments.

No. 1675.—A sandy soil with little humus, from the test farm of the North Carolina department of agriculture at Red Springs.

Nos. 1676, 1677, 1678, and 1679 are soils with little humus, from the Rhode Island station. No. 1676 is from plat 23 and No. 1677 from plat 25, both of which have received ammonium sulphate for several years, the former without and the latter with the application of lime. No. 1678 is from plat 27 and No. 1679 from plat 29, both of which have received sodium nitrate for several years, the former without and the latter with the application of lime.

No. 1680.—Sandy soil with little humus, from the test farm of the North Carolina department of agriculture at Tarboro.

The following table shows the amount of nitrates present at the end of three weeks in samples to which no nitrogenous matter was added in the experiment:

Nitrates formed in blank experiments.

No.	Without calcium carbonate.	With calcium carbonate.	No.	Without calcium carbonate.	With calcium carbonate.
	Mg.	*Mg.*		*Mg.*	*Mg.*
1667	10.4	14.0	1676	18.5	([a])
1668	2.5	8.4	1677	14.5	([a])
1669	2.2	([a])	1678	7.3	([a])
1670	4.2	6.5	1679	3.1	([a])
1674	16.3	17.7	1680	1.8	5.0
1675	1.2	([a])			

[a] Not determined.

The table given below shows the precentages of nitrogen which were nitrified under the conditions stated.

Nitrogen converted to nitrates.

Soil number.	Moisture, average.	Temperature.		Nitrogenous material used.	Nitrified.	
		Extreme.	Average.		Without calcium carbonate.	With calcium carbonate.
	Per cent.	°*C.*	°*C.*		*Per cent.*	*Per cent.*
1667	10.5	25-30	27	Ammonium sulphate	7.2	57.2
				Cotton-seed meal	32.4	13.6
				One-half ammonium sulphate	....	71.8
				One-half cotton-seed meal	....	43.0
				Ammonium sulphate and cotton-seed meal	....	22.3
1668	13.0	25-30	27	Ammonium sulphate	0.6	11.6
				Cotton-seed meal	−0.5	−1.1
				One-half ammonium sulphate	....	43.2
1669	13.0	25-30	27	Ammonium sulphate	0.2	15.8
				Cotton-seed meal	17.3	41.5
1670	15.0	23-28	26	Ammonium sulphate	2.2	45.7
				Cotton-seed meal	14.8	32.6
				Ammonium sulphate and cotton-seed meal	0.5	....
1671	10.75	20-26	22	Ammonium sulphate	2.1	2.3
				Cotton-seed meal	3.4	4.9
				One-half ammonium sulphate	....	16.5
				One-half cotton-seed meal	....	10.9
				Ammonium sulphate and cotton-seed meal	1.3	....

Nitrogen converted to nitrates—Continued.

Soil number.	Moisture, average.	Temperature.		Nitrogenous material used.	Nitrified.	
		Extreme.	Average.		Without calcium carbonate.	With calcium carbonate.
	Per cent.	°*C.*	°*C.*		*Per cent.*	*Per cent.*
1675	8.0	19–24	20	Ammonium sulphate	None.	None.
				Cotton-seed meal	None.	None.
1676	12.2	18–23	19	Ammonium sulphate	−0.6	1.5
				Cotton-seed meal	−1.9	−1.3
				One-half ammonium sulphate	....	1.5
				One-half cotton-seed meal	....	0.6
1677	11.7	18–23	19	Ammonium sulphate	1.0	5.6
				Cotton-seed meal	1.8	6.2
1678	12.1	18–23	19	Ammonium sulphate	−0.3	0.6
				Cotton-seed meal	−0.1	−0.3
1679	12.5	18–23	19	Ammonium sulphate	1.2	3.0
				Cotton-seed meal	2.9	10.6
1680	9.5	19–24	20	Ammonium sulphate	−0.1	−1.3
				Cotton-seed meal	1.3	−0.8
				One-half ammonium sulphate	....	−2.3
				One-half cotton-seed meal	....	3.2

The following observations are made on the above tables:

(1) Calcium carbonate increases the rate of nitrification of nitrogenous matter already in the soil, and also of ammonium sulphate and of cotton-seed meal added, in those cases where nitrification took place at all.

(2) The rate of nitrification was increased when a smaller percentage of nitrogenous material was added to the soil.

(3) Some soils do not nitrify cotton-seed meal either with or without the aid of calcium carbonate.

(4) Some soils do not nitrify ammonium sulphate either with or without the aid of calcium carbonate.

(5) Some soils which do not nitrify ammonium sulphate nitrify it upon the addition of calcium carbonate.

(6) Hydrogen sulphid is not generated when ammonium sulphate is mixed with soils.

(7) In all soils used by us which nitrify ammonium sulphate and cotton-seed meal, nitrification of cotton-seed meal took place more rapidly than the nitrification of ammonium sulphate in the absence of calcium carbonate.

(8) In soils which nitrify both materials, sometimes in the presence of calcium carbonate, cotton-seed meal is nitrified more rapidly and sometimes less rapidly than ammonium sulphate.

(9) Soils which in their natural state are capable of nitrifying cotton-seed meal lose this power in the presence of ammonium sulphate in case calcium carbonate is absent, and even where calcium carbonate is present one of these materials exercises a retarding action upon the nitrification of the other.

(10) Some soils to which ammonium sulphate and lime have been added for a series of years nitrify ammonium sulphate more rapidly than where there has been no application of ammonium sulphate.

CONCLUSIONS.

From the above observation the following explanations which were previously offered by us (Bul. No. 176, North Carolina station) seem to be sustained:

(1) Ammonium sulphate in some cases hinders the action of nitrifying organisms. In soil No. 1670, 14.8 per cent of nitrogen in cotton-seed meal was nitrified, but upon the addition of ammonium sulphate only 1.3 per cent of the nitrogen was nitrified. In No. 1674 similar results are observed.

(2) In the nitrification of ammonium sulphate, sulphuric acid or nitric acid or both are formed and hinder further nitrification unless neutralized. In soil No. 1668,

7.2 per cent of the nitrogen is nitrified without calcium carbonate, and with calcium carbonate 57.2 per cent. The results in soils Nos. 1669 and 1670 are equally as marked.

(3) Soils differ as to the nitrifying organisms which they contain, some being capable of nitrifying readily both ammonium sulphate and cotton-seed meal; some nitrifying cotton-seed meal more rapidly and some less readily than ammonium sulphate; some nitrifying ammonium sulphate, but not cotton-seed meal, and some being capable of nitrifying either ammonium sulphate or cotton-seed meal.

The following additional conclusions seem justified:

(4) The relative number of organisms in any soil capable of nitrifying ammonium sulphate may be increased by the continued addition of this substance and lime if such germs were originally present.

(5) Calcium carbonate is very helpful in the nitrification of all substances.

The authors wish to express their thanks to Director H. J. Wheeler, of Rhode Island; State Chemist B. W. Kilgore, of North Carolina; Agriculturist W. P. Brooks, of Massachusetts, and Chemist H. K. Miller, of Florida, for their kindness in furnishing samples of soils used in this work.

Mr. Frear. There is one source of error in the determination of humus or matière noire by the official method of which I have seen no mention. As is well known, the method consists in the solution of the humus freed from basic combination by previous extraction with dilute hydrochloric acid in a dilute solution of ammonia. Separation of the ammoniacal solution from insoluble solids is wrought by aid of simple filtration, and from other soluble solids by weighing the total solid residue left upon evaporation and careful drying, igniting it, again weighing the incombustible residue, and estimating the humus as equivalent to the loss by ignition.

The fallacy of the assumption that simple filtration, drying under ordinary conditions in a water-oven, and ignition are sufficient to separate the ammonia-soluble humus from the other soil constituents was very interestingly brought to notice in the course of analyses of certain deep-red Cuban tobacco soils, made under my direction by Mr. C. P. Beistle in my laboratory during the past year.

Mechanical analysis of the ignited soil showed that from 50 to 65 per cent by weight was composed of particles between 0.5 and 0.05 mm in diameter; that the soil, in its present condition of cultivation, behaves like a medium to fine sand. It contains 15 to 18 per cent of particles less than 0.01 mm in size.

The last washings proved, however, to contain much very fine material. The beaker containing the fine silt and clay stood for more than four months undisturbed, and was yet highly discolored with suspended material.

The soil is formed by the weathering of certain chalky formations in the southern part of the province of Habana. Yet the quantity of lime is quite small, 0.27 to 0.37 per cent.

When these soils were treated with dilute ammonia the deflocculating influence of this agent was remarkably exhibited. Nearly the entire body of the soil became finely subdivided, and passed through single and double filters and single and double hardened S. & S. filters. The ignited residues from the "humus" determination, even when hardened filters were employed, constituted from 60 to 83 per cent of the original weight of air-dry soil. Only upon use of a Pasteur filter were we able to secure a clear solution. The filter was covered with dark, colloidal matter. No data have been accumulated to show the effect of such filtration upon the humus in solution; therefore the results of analysis of the filtrate can not be regarded as trustworthy.

The mere fact that fine particles of mineral matter pass the filter with the humus would be of no consequence if the material were such as to undergo no loss upon ignition; but, in fact, the clay particles which most readily pass the filter are commonly the most highly hydrated mineral materials of the soil. These Cuban soils lost, when ignited, from 17 to 20 per cent of their "water-free" weight. They contained of organic carbon in the surface soils 2.5 to 2.8 per cent, in the subsoil 1.3 to 2.1 per cent. These quantities of carbon correspond to 4 to 5 per cent of humus in the surface soils and to 2.5 to 3.5 per cent in the subsoils. Therefore the principal loss upon ignition was due to elimination of water of combination. The correctness of this conclusion was proven by the determination of water in the combustion products.

The percentages of "humus" extracted by ammonia were, as determined by the official method, from 11.25 to 14.62 per cent of the water-free soil; combustion of several combined residues gave only 1.65 per cent of carbon, corresponding to about 3 per cent of humus. The principal part of the loss by ignition is due, in other words, to the water of combination given up by the hydrated minerals that passed the filter with the humus.

Doubtless the soil upon which this result was obtained is very unusual. Nevertheless, it leaves a serious question as to the value of results obtained by the present method when applied to clay soils. It is desirable that actual determinations of carbon, and possibly hydrogen, be made upon the humus residues as checks upon the analytical work in the case of such soils.

The PRESIDENT. The secretary has some announcements to make before we adjourn.

Mr. WILEY. I have a communication here from the secretary of the Cosmos Club inviting us to meet at the club this afternoon.

(Mr. Wiley at this point read the letter received.)

The PRESIDENT. The Chair will announce the committees provided for by this morning's meeting.

COMMITTEES ON RECOMMENDATIONS OF REFEREES.

A. Phosphoric acid, potash, nitrogen, soils, ash, and insecticides—Messrs. Huston, Ross, Hopkins, Haywood, and Hite.

B. Dairy products, foods, feeding stuffs, and tannin—Messrs. Winton, Penny, Kilgore, Patrick, and Krug.

C. Liquors and food adulterations—Messrs. Bigelow, Caldwell, Withers, Woods, and Munson.

Mr. Kilgore. I would like to offer this resolution:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

The meeting adjourned at 1.30 p. m.

THURSDAY—AFTERNOON SESSION.

President Van Slyke called the meeting to order at 1.25 p. m.

The President. There is an additional paper to be presented on soils, and that will now be in order.

Mr. Cameron. The paper referred to is entitled "Chemical Examination of Alkali Soils," by Atherton Seidell, and is one of several papers published collectively under the title of "Solution Studies of Salts Occurring in Alkali Soils," as Bulletin 18 of the Division of Soils, United States Department of Agriculture. It will not be necessary to read it at this time, but I move that the paper and the method outlined therein be referred to the committee on recommendations.

The President. The motion has been made that this paper be referred to the committee on recommendations. It is carried.

Is there any business that anyone wishes to bring forward at this time, before we go on with the referee's reports?

Mr. Veitch. Before we leave the question of soils I have a resolution that I would like to offer. We find the methods very unsatisfactory, and to overcome that condition and get the matter before the association I would like to introduce the following resolution:

Resolved, That the policy of the association as to its way of investigating analytical methods for the determination of available plant food be changed. That, in place of asking the cooperation of all chemists in the comparative study of two or more methods on a furnished set of samples, each of those chemists who are willing to cooperate in the work be asked by the referee to take up such a line of soil study, by any method he may choose, upon known soils of his own State or upon other known samples, the avowed purpose of such a study being the working out of a chemical method for determining the available plant food of soils.

Further, That those who engage in this work shall keep the referee informed of their lines of investigation, shall furnish him with a summary of their results (to be presented by him to the association at the regular meetings), and shall, from time to time as the occasion warrants, present to the association the details of their work.

The President. In my judgment, that resolution would more properly go to the special committee on recommendations of referees, and if there is no objection it will be passed on and submitted to that committee, of which Mr. Huston is chairman.

Mr. Hopkins. If it is in order, before we leave the subject of soils I would like to make a few remarks regarding the determination of potash by our official method.

SEPARATION OF ALKALIES IN SOIL ANALYSIS BY THE OFFICIAL METHOD.

By C. G. Hopkins.

In connection with our soil investigations in Illinois, where we are making a large number of analyses, we have had some difficulty in obtaining accurate results by the official method, especially in the separation of the alkalies.

After the precipitation of iron, aluminum, magnesium, etc., by means of barium hydroxid, and of calcium and barium by means of ammonium carbonate, exactly according to the official method (a second precipitation with ammonia and ammonium carbonate having been made), the final filtrate (which is ordinarily simply evaporated to dryness and ignited to secure the weight of the alkaline chlorids) was found to be by no means free from barium.

Even after a third precipitation with ammonia and ammonium carbonate very appreciable amounts of barium remain in the filtrate. Ten separate determinations of the barium remaining in this filtrate (after three precipitations with ammonia and ammonium carbonate) gave the following amounts of barium sulphate:

	Gram.		Gram.
1	0.0065	6	0.0080
2	.0083	7	.0113
3	.0088	8	.0107
4	.0075	9	.0099
5	.0061	10	.0111

It will be seen that this error, in the determination of the alkalies by the official method, is one of considerable importance.

We are now using a modification of the official method in our laboratories, in which we precipitate the last traces of barium by the addition of ammonium sulphate. A sufficient quantity of ammonium sulphate is added so that in the ignition of the residue from the evaporation of the filtrate the alkalies are converted to sulphates and are thus rendered less liable to loss in ignition, an additional advantage possessed by our modification. With the help of Mr. J. H. Pettit, one of our chemists, we are accumulating a considerable amount of data relating to this modified method for separating the alkalies in soil analysis, and now expect to have the matter ready for publication in a few weeks.

I may add that in conversation with others who are familiar with the details of soil analysis I have learned that the official method for the separation of alkalies is commonly regarded as inaccurate, and I suggest that this proposed modification be referred to the proper committee on recommendations.

The President. The paper will be so referred if there is no objection. Is there anything more on soils?

The following letter from Mr. Peter, inclosing analytical data obtained by Mr. Beatty, was then presented:

Lexington, Ky., *November 7, 1901.*

My Dear Sir: Inclosed herewith please find results on the Association of Official Agricultural Chemists soil samples obtained in this laboratory by Mr. L. O. Beatty, one of our assistant chemists. We did not have time or material to duplicate the results, but Mr. Beatty has been engaged in this kind of work and the figures are worthy of confidence. The method used in determining the K_2O was somewhat different from that of the referee. An aliquot of the solution corresponding to 100 grams of air-dry soil was evaporated to dryness to separate silica; taken up again with hydrochloric acid and water, filtered, and the solution precipitated with ammonium hydrate and oxalate of ammonia, filtered, and washed. The filtrate and washings were evaporated to dryness, and the ammonia salts expelled by careful heating. The potassium was then determined in the usual way by platinum chlorid.

Very respectfully,

Alfred M. Peter.

Mr. F. P. Veitch,
Associate Referee, Washington, D. C.

Results of work by Mr. L. O. Beatty, Kentucky Agricultural Experiment Station, on the soil samples for Association of Official Agricultural Chemists.

Laboratory number	8594.	8595.	8596.	8597.	8598.
Referee's number	1.	2.	3.	4.	5.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Moisture, drying in water-oven	1.60	1.04	1.89	0.93	1.09
Combined water, organic matter, etc	6.48	2.77	7.11	4.88	5.40
Humus, official method	1.81	.32	2.56	1.55	1.98
K_2O by N/5 HCl, 5 hours' digestion at 40° C.; precipitated out the iron, alumina, and lime with ammonia and ammonium oxalate, and expelled ammonium salts by heat	.1194	.0215	.0152	.0123	.0168
K_2O same, except the temperature of digestion reached 50°	.1120	....	....	....	....
K_2O by N/5 HNO_3, digested 5 hours at the room temperature (about 24°); iron, etc., separated as above	.0838	.0181	.0133	.0106	.0127
P_2O_5 by N/5 HCl, digested 5 hours at 40° C.; determined volumetrically	.1760	.0686	.1223	.0465	.0215
P_2O_5 by N/5 HNO_3, digested 5 hours at room temperature; determined volumetrically	.1688	.0650	.1088	.0275	.0134

The President. Next in order is the report on ash. The referee is not present. Is the referee on foods and feeding stuffs ready to report?

REPORT ON CATTLE FEEDS.

By William H. Krug, *Referee.*

A circular letter requesting cooperation in the work on feeding stuffs brought favorable replies from 16 chemists, to whom the following samples were sent:

No. 1. Wheat.
No. 2. Bran.
No. 3. Clover seed.

The suggestions embodied in the report for 1900 were made the basis for the following recommendations as to lines of research which appeared worthy of attention:

I. *Moisture.*—Determine the moisture in all samples by the following methods:

a. Twenty-four hours in a water bath at a temperature at which water boils in your locality. State this temperature in your report.

b. To constant weight in a water bath as in *a.*

c. To constant weight in an air bath at 100° C., or if constant weight can not be obtained, until two consecutive dryings of an hour each show a loss of less than 2 mg. State the method pursued.

d. As prescribed in Bulletin 46, revised edition (U. S. Department of Agriculture, Division of Chemistry), page 23, II, 2.

e. To constant weight as in *d.*

f. To constant weight in vacuo at 70° C.

Starch—

a. Determine the starch in samples 1 and 2.

b. Collect the cuprous oxide on a tared Gooch crucible and weigh as such, calculating it as Cu_2O.

c. Determine the solvent action of the diastase and water on the pentosan by evaporating an aliquot part of the solution to a small volume, and distilling with 12 per cent hydrochloric acid.

Pentosan—

a. Determine the pentosan in samples 1 and 2.

b. Determine the effect which variations in the time of standing after precipitation with phloroglucol have on the quantity of phloroglucid obtained. Make a series of determinations in which the precipitated solution is allowed to stand at least twenty hours.

c. Determine the effect of the rapidity of distillation on the quantity of furfural obtained by regulating the burner so as to distill over 20, 25, 30, etc., cc per ten minutes.

Galactan.—Determine the galactan in sample 3.

There has been an evident lack of interest in the work this year, and only four reports have been received.

THE ESTIMATION OF MOISTURE.

The results reported on last year's samples showed such wide and startling variations that it appeared desirable to investigate the causes which led to such errors in a determination apparently simple and easy of execution. Various methods of drying were suggested, and the results are given in the following table:

TABLE I.—*Moisture determinations.*

Analyst.	Method of drying.	Sample No. 1.	Sample No. 2.	Sample No. 3.
		Per cent.	*Per cent.*	*Per cent.*
G. S. Fraps, North Carolina	Caldwell water oven and Caldwell tubes, 7 hours.	11.81	11.34	....
F. S. Shiver, South Carolina	Water oven, 24 hours	11.15	10.56	6.99
T. M. Price, Maryland	....do	10.38	10.58	6.67
F. S. Shiver, South Carolina	Water oven, constant weight, 99° C.	11.15	10.54	7.31
E. B. Holland, Massachusetts	Glycerin and water oven, about 100° C., 29 hours.	11.06	10.05	7.07
W. H. Krug, U. S. Department of Agriculture.	Water oven, 98° C., 26 hours	....	9.84	....
J. H. Norton, U. S. Department of Agriculture.	In vacuo, 70° C., 10 hours	....	9.56	....
W. H. Krug, U. S. Department of Agriculture.	In vacuo, 70° C., 15 hours	10.84	10.19	6.65
J. H. Norton, U. S. Department of Agriculture.	Air bath, 100° C., 7 hours	....	9.96	....
W. H. Krug, U. S. Department of Agriculture.	Air bath, 100° C., 5 hours	....	10.14	....
Mean of all determinations		11.06	10.28	6.94

Mr. Fraps reports that when the samples were dried five hours in a Caldwell oven, using Caldwell tubes, the following results were obtained: No. 1, 11.75 per cent; No. 2, 11.07 per cent. Two hours additional gave the results found in the table, showing that there was only a slight loss in weight after five hours.

The figures presented this year show a marked improvement when compared with those reported in 1900, but differences still exist, and these are most marked when we compare the results obtained in vacuo at 70° C. with those obtained by drying in air, the former being almost uniformly lower, although the drying was continued until no further loss occurred, the time required to reach this point being in every case fifteen hours. It is evident that where materials of this kind are dried in air, this being the method most generally followed, the final result does not represent the absolute moisture present, but is increased by losses due to exposure to the air at the temperature employed. The official methods for the analysis of foods direct that the material shall be dried for five hours, at the temperature of boiling water, in a current of dry hydrogen, or in vacuo. Neither of these methods has been generally adopted, as most laboratories are not equipped with the necessary facilities and are thus forced to use some other method. In such a case the ordinary air bath should, however, never be used, as it is practically impossible to control the temperature, even on the same shelf, whereas this is easily accomplished when a water oven is employed. It being generally acknowledged that the losses noted when a material is dried in air are not wholly due to moisture, it is naturally not feasible to carry the drying to the point where a gain in weight takes place, as this requires considerable time and is of no advantage, since it does not yield an absolute value. A time limit must therefore be fixed, and in view of the fact that in most cases the moisture determinations are made by drying the substance in air, it seems advisable to incorporate certain directions in the official methods, and your referee would suggest that we adopt, as optional in the case of feeding stuffs, the method given for sugars, which is as follows:

> Dry from 2 to 5 grams in a flat dish (nickel, platinum, or aluminum), at the temperature of boiling water, for ten hours; cool in a desiccator and weigh; return to the oven and dry for an hour. If on weighing there be only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until the loss of water in one hour is not great.

THE ESTIMATION OF STARCH.

So little work has been done on the diastase method this year that it is impossible to draw conclusions or to make any suggestions as to changes. The results are given in Table II. Owing to the different methods used to weigh the copper, the results are not averaged.

TABLE II.—*Starch determinations.*

Analyst.	Sample 1.		Sample 2.	
	In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
F. S. Shiver, South Carolina	[a] 53.68	60.42	[a] 8.57	9.58
J. H. Norton, U. S. Department of Agriculture	[b] 56.55	63.43	[b] 13.95	15.53
Do	[c] 56.03	62.84	[c] 13.50	15.03

[a] Copper weighed as CuO. [b] Copper weighed as Cu_2O. [c] Copper weighed as Cu.

The figures reported by Mr. Norton represent in each case an average of six determinations, and the amount of copper obtained by directly weighing the oxid and assuming its composition to be Cu_2O was in every instance higher than when the oxid was dissolved and the copper obtained electrolytically, showing thereby that the red oxid is not uniform in composition and affords but an uncertain basis for calculation. This is in accord with the statements made in the literature from time to time by European chemists.

THE ESTIMATION OF PENTOSAN.

The results are given in the following tables:

TABLE III.—*Pentosan determinations.*

Analyst.	Sample 1.		Sample 2.	
	In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
F. S. Shiver, South Carolina	7.57	8.52	25.12	28.08
E. B. Holland, Massachusetts	7.08	7.96	24.71	27.47
G. S. Fraps, North Carolina	6.30	7.14	23.00	25.97
T. M. Price, Maryland	7.83	8.74	26.01	29.09
J. H. Norton, U. S. Department of Agriculture	6.76	7.58	25.20	28.06
Average	7.11	7.99	24.81	27.73

COMMENTS.

G. S. Fraps.—It is impossible to dry the phloroglucid precipitate to constant weight by heating three to four hours, especially if it is large. The following figures were obtained by drying four hours, and continuing the drying:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.
	Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*
Weight, 4 hours	0.2135	0.2227	0.8932	1.1741	1.0786
Weight, 6 hours	.2037	.2127	.7194	.7443	.6621
Loss	.0098	.0100	.1738	.4298	.4165
Eight hours					.6563
Loss					.0058

It would be easy to give a number of examples. I usually dry six hours, weigh, and then dry two more hours.

A determination of furaloid[a] was made in the distillates by redistilling them. The crude furfural from sample No. 1 contained 19 per cent, No. 2, 12 per cent furaloid.

Mr. Holland reported in detail the individual determinations, showing a very close agreement. He determined the pentosan in both samples, with and without previous extraction with ether, and obtained the following figures: No. 1, not extracted, 7.18 per cent; extracted, 6.98 per cent; No. 2, not extracted, 24.69 per cent; extracted, 24.73 per cent. These data indicate that previous extraction with ether is not necessary, except, possibly, in the case of substances containing a very high percentage of fat.

In the directions sent out with the samples your referee suggested that experiments be made to determine the effect which the time of standing after precipitation has on the yield of pentosan. Considerable work has been done on this point, and the results are given in the following table:

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[a] See Am. Chem. Jour., 1901, **25**, 502.

TABLE IV.—*Influence of time precipitate is allowed to stand on yield of pentosan.*

Analyst.	Time of distillation per 30 cc.	Time precipitate was allowed to stand.	Sample No. 1.		Sample No. 2.	
			In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Minutes.	*Hours.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
T. M. Price, Maryland	10	1	7.83	8.74	....	....
Do	10	5	7.83	8.74	....	....
J. H. Norton, U. S. Department of Agriculture	10	16	....	....	24.90	27.73
Do	10	20	6.76	7.58	25.20	28.06
F. S. Shiver, South Carolina	10	20	7.57	8.52	25.12	28.08
G. S. Fraps, North Carolina	10	20	6.30	7.14	23.00	25.97
E. B. Holland, Massachusetts	9.5	20+	7.18	8.07	....	....
Do	10+	20+	....	....	24.69	27.45
Do	10	20+	....	....	24.73	27.49
Average	....	....	6.95	7.83	24.55	27.41
E. B. Holland, Massachusetts	10.25	21	6.98	7.85	....	....
J. H. Norton, U. S. Department of Agriculture	10	24	....	....	25.68	28.59
T. M. Price, Maryland	10	24	7.83	8.74	26.01	29.09
Average	....	....	....	....	25.85	28.84
T. M. Price, Maryland	10	48	7.83	8.74	....	....

The considerable variations which exist between the individual data obtained by a supposedly uniform method of working render it rather difficult to base any definite conclusions on these results. They can only be accepted as an indication that an increase in the time of standing from twenty hours (as now directed) to twenty-four hours results in a higher yield of phloroglucid. Further confirmation is, however, necessary before any such change in the present method is warranted. In connection with this point we must, furthermore, consider the fact discovered by Mr. Fraps, that the distillate always contains a substance which separates as a black precipitate on standing, and one or more substances which are not furfural, but are precipitated by phloroglucol. He has given the latter the name "furaloid," and has published an interesting paper on the subject in the American Chemical Journal, volume 25, No. 6, page 501. His summary is as follows:

> The distillate from vegetable materials with hydrochloric acid may contain, besides other things, fatty acids, a substance which separates as a black precipitate if the vessel is allowed to stand, and a substance (or substances) which is destroyed by distilling again (furaloid). All three of these may appear in the phloroglucol precipitate.
>
> The furaloid-yielding bodies were found in all the materials treated, the crude furfural in the hydrochloric acid distillate containing from 7 to 23 per cent of furaloid.
>
> The furaloid-yielding bodies are hydrolyzed by boiling 1.25 per cent sulphuric acid, and lost in the subsequent evaporation. They are digested to a greater extent than the total pentosans in the one sample of timothy hay excrement tested.
>
> The furaloid is not formed by the action of the hydrochloric acid on the pentose sugars.

While there are thus several sources of error in the present method, in so far as its value as a means of determining the actual amount of pentosans present is concerned, Mr. Fraps does not believe it expedient to vary the method so as to permit the determination of the furfural and furaloid separately, as they can be very conveniently grouped together. An experiment made by him showed that the furaloid-yielding bodies are somewhat more digestible than the pentosan.

Your referee further suggested that experiments be made to determine the influence of the time of distillation on the yield of pentosan. The results are given in Table V.

TABLE V.—*Influence of time of distillation on yield of pentosan.*

Analyst.	Time of distillation per 30 cc.	Time precipitate was allowed to stand.	Sample No. 1.		Sample No. 2.	
			In original sample.	Calculated to water-free basis.	In original sample.	Calculated to water-free basis.
	Minutes.	*Hours.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. H. Norton, U. S. Department of Agriculture	5	20			21.33	23.75
Do	10	20	6.76	7.58	25.20	28.06
E. B. Holland, Massachusetts	9.5	20+	7.18	8.07		
Do	10.25	21	6.98	7.85		
Do	10+	20+			24.69	27.45
Do	10	20+			24.73	27.49
Average			6.98	7.83	24.87	27.67
J. H. Norton, U. S. Department of Agriculture	15	20			24.84	27.66
E. B. Holland, Massachusetts	15	22+	6.83	7.68		
Do	15	20+			22.56	25.08
T. M. Price, Maryland	15	24			24.80	27.73
Average					24.07	26.82

It is very evident that the time of distillation at present prescribed by this method is best, as it gives the highest results. The primary object of the investigation was to determine if the time of distillation could be shortened, as this feature of the method is tedious and time-consuming. Mr. Norton's results in a series of determinations showed conclusively that when the time of distillation was reduced to five minutes for every 30 cc the yield of pentosan was decreased as much as 4 per cent. Neither does an increase of the time to fifteen minutes possess any advantages, the results indicating that this also causes a slight decrease in the yield.

E. Kröber[a] has recently published a long and exhaustive study of the phloroglucol method, and his conclusions may be briefly summarized as follows:

(1) That the result is not influenced by the length of time the precipitates stand.

(2) That the precipitate is best collected in a gooch.

(3) That the wash water must be added in small portions and always before the precipitate in the gooch has become too dry.

(4) That the precipitate must not contain hydrochloric acid, as the presence of the acid leads to high results, owing to oxidation.

(5) That the presence of diresorcol in the phloroglucol does not influence the result.

(6) That about twice as much phloroglucol must be added as there is furfural present.

(7) That the phloroglucol must previously be dissolved in dilute hydrochloric acid (sp. gr. =1.06).

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[a] Journal für Landwirtschaft, 48, 537.

(8) That a uniform time of drying is indicated (four hours at 98.5° to 100° C.), as the last traces of water are very difficult to remove and eventual oxidation will cause errors.

(9) That the gooch must be weighed in a weighing bottle, as the phloroglucid is very hygroscopic.

The author further recommends that the quantity of substance taken for distillation be such that the weight of the phloroglucid shall not exceed 0.300 gram.

The chief difference between these conclusions and the results obtained by the chemists of this association is found in the statement that the presence of diresorcol does not affect the final result. In support of this the author gives a series of results obtained by precipitating a solution of furfural with pure and impure phloroglucol. The averages are as follows:

	Weight of phloroglucid.	
	Series 1.	Series 2.
	Gram.	*Gram.*
Pure phloroglucol	0.08755	0.04035
Impure phloroglucol	.08760	.04055

This point is, however, not of much importance, as the official method prescribes a convenient process for the purification of the phloroglucol. The suggestions made by Kröber with reference to the precautions to be taken in drying and weighing are important, and your referee believes it advisable to incorporate them in our method.

THE ESTIMATION OF GALACTAN.

The results obtained are given in the following table.

TABLE VI.—*Galactan determinations.*

Analyst.	Sample 3.	
	In original sample.	Calculated to water-free basis.
	Per cent.	*Per cent.*
F. S. Shiver, South Carolina	5.12	5.52
E. B. Holland, Massachusetts	5.05	5.43
T. M. Price, Maryland	3.07	3.29
J. H. Norton, U. S. Department of Agriculture	5.73	6.14
Average	4.74	5.09

With the exception of the figures reported by Mr. Price, these results are satisfactory. The conversion of galactan into mucic acid is never complete in practice and it is therefore impossible to obtain the theoretical yield. This fact in itself is such a possible source of error that very closely agreeing results can not be expected.

RECOMMENDATIONS.

The recommendations this year deal mainly with minor changes tending toward more exact methods.

Moisture.—It is recommended that the method used for the drying of sugars be adopted as optional for the drying of feeding stuffs.

Phloroglucol method.—It is recommended that—

a. The first line be changed so as to read, "A quantity of the material, chosen so that the weight of the phlorogucid obtained shall not exceed 0.300 gram, is placed in a flask." The use of 3 grams of material, as now specified, often leads to such large quantities of phloroglucid that filtration, subsequent washing, and drying of the precipitate are very difficult. By basing the quantity of material on a definite weight of phloroglucid which is small enough to present no difficulties, this will be avoided.

b. In the seventh line the words "by means of a separatory funnel and" be inserted after "added." This insertion is merely explanatory, as there appears to have been some doubt in the past as to the form of apparatus which should be used.

c. In the seventeenth line the words "from three to" be stricken out. Your referee believes that the fixation of a time limit in connection with the first recommendation will tend to give more concordant results.

d. In the eighteenth line the words "in a weighing bottle" be inserted after "weighed." The hygroscopic nature of the phloroglucid renders the necessity of this precaution self-evident.

Galactan method.—It is recommended that—

a. In the twelfth line the word "gently" be stricken out, the words "at 80° C" be inserted after "bath," and the words "with constant stirring" after "minutes." The directions are rather indefinite at this point, and it was thought advisable to fix a definite temperature of digestion. In the absence of definite information, the temperature used in the laboratory of the Hatch Experiment Station was chosen. The object of the digestion with the ammonium carbonate solution is to convert the mucic acid into ammonium mucate, and this is naturally facilitated by stirring the mixture constantly.

b. In the seventeenth line the words "avoiding unnecessary heating, which causes decomposition," be inserted after "bath." This insertion is a necessary precaution, as the residue obtained on evaporation of the filtrate rapidly darkens when subjected to further heating.

c. In the twenty-first line the words "three hours" be substituted for "a short time." A definite time of drying appears preferable to the directions now given.

The PRESIDENT. Any paper relating to this subject is now in order.

THE DETERMINATION OF PENTOSAN-FREE CRUDE FIBER.

By G. S. FRAPS.

Until a few years ago, the only demand made upon the method of determining crude fiber was that the crude fiber should not contain appreciable quantities of nitrogenous bodies. A method was chosen which would give a crude fiber having a minimum nitrogen content, always bearing in mind other considerations, as its convenience, accuracy, and ability to give concordant results in the hands of different analysts.

In the last few years a determination has been introduced into our scheme of analysis for foods which compels us to demand more of the crude fiber determination. The introduction of the pentosan determination compels us to choose between two courses—to require that the crude fiber should not contain appreciable quantities of pentosans, or to make two determinations of pentosans when necessary—one of total pentosans and one of pentosans in crude fiber.

Does the crude fiber prepared by the official method contain pentosans? This question has been answered in the affirmative. The crude fiber contains pentosans, often in considerable quantity, as is shown by the following figures selected from the large number available:

TABLE I.—*Pentosans in 100 parts of crude fiber.*

	Per cent.
J. König, Analyst, **23**, 47 (1898):	
Rye straw	18.4
Pea straw	15.0
Clover hay	12.7
Wheat bran	2.6
Bulletin 172, N. C. Station (1900):	
Timothy hay	14.4
Crabgrass hay	13.4
Green rape	7.7
Rice bran	8.3

Since the official method does not yield a pentosan-free crude fiber, we have the choice of two alternatives: To adhere to this method and determine pentosans in crude fiber when necessary, or to seek for a new method by which a pentosan-free crude fiber may be prepared.

KÖNIG'S METHOD.

In an article read before this body last year the writer directed attention to the method of J. König for determining pentosan-free crude fiber. This consists in boiling the material with glycerol containing sulphuric acid, the boiling point of the glycerol being 131° to 133° C.[a]

Kellner, Herring, and Zähn tested this method, and decided that it is an improvement over the one in present use, both in speed and accuracy.[b]

C. Beck[c] did not decide quite so favorably. He found it difficult to keep the temperature of the glycerol between 131° and 133°, and consequently obtained results which varied somewhat. He concluded that the method may be particularly useful for fodders, but for finely ground grain feeds the other method is more reliable.

König replied that the method was better adapted for grain feeds. The temperature could easily be kept at 131° to 133° if glycerol of exactly 1.229 specific gravity is used, and the boiling conducted so gently that only a few drops of water collect in the condenser.

During the past summer the writer undertook some study of König's method. The full description of the method, with such precautions and observations as seem necessary, is as follows:

DETERMINATION OF CRUDE FIBER.

(*a*) *Glycerol-sulphuric acid.*—Determine the specific gravity of the glycerol by means of a picnometer or specific-gravity balance. A hydrometer can not be used, as on glycerol its readings would not be correct. The per cent of glycerol may be calculated by the following table:

Determination of glycerol by specific gravity.

Specific gravity at 15.5°.	Glycerol.	Specific gravity at 15.5°.	Glycerol.	Specific gravity at 15.5°.	Glycerol.
	Per cent.		*Per cent.*		*Per cent.*
1.2674	100	1.2540	95	1.2406	90
1.2647	99	1.2513	94	1.2380	89
1.2620	98	1.2486	93	1.2353	88
1.2594	97	1.2460	92	1.2327	87
1.2567	96	1.2433	91	1.2300	86

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[a] Analyst, **23**, 47, or Exp. Station Record, **9**, 1021.

[b] Exp. Station Record, **11**, 705.

[c] Ibid., **12**, 611.

Should the temperature of the specific-gravity determination not be made at 15.5°, a correction may be made by adding 0.00058 for each degree above that temperature.

Dilute the glycerol to 86 per cent, or 1.230 specific gravity, and make up a solution containing 20 cc sulphuric acid (1.84 specific gravity) per liter of the glycerol-acid mixture.

(*b*) *The determination.*—Place 3 grams of substance in a 500 cc Erlenmeyer flask, add 200 cc glycerol-sulphuric acid, and connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Heat to boiling, and boil very gently for an hour, shaking the flask from time to time, so as to wash down any particles. The boiling should take place in such a manner that only a few drops of water are condensed. Prepare a thin layer of asbestos in a 2-inch Hirsch funnel, and on this place a perforated platinum disk. Filter the glycerol on this, using a suction pump. The mixture should be shaken well before it is poured on the filter, and each flask should have the full benefit of the suction pump when the filtration starts, until the glycerol begins to come through. Wash with hot water, with alcohol, and with a mixture of equal volumes of alcohol and ether. The alcohol and ether remove, besides fats, certain products not soluble in water, formed by the action of the glycerol acid. Transfer to a platinum dish, dry, weigh, and incinerate completely. The loss in weight is crude fiber.

NECESSARY PRECAUTIONS.

A primary necessity is to have the glycerol of the required density. A comparatively slight variation in density will cause an increase or decrease in boiling point, with a corresponding decrease or increase in the crude fiber.

On account of the nature of glycerol, it is necessary to filter it as hot as possible and before it cools. Cold glycerol, even if diluted with water, filters very slowly indeed. The following method of filtration is recommended as the best, after trying several ways:

(1) Dilution with water and filtration while hot, as recommended by König. It seems impossible to get the liquid through the filter sometimes when this method is used. It always takes longer than the method recommended, if properly carried out.

(2) Dilution with alcohol. Filtration takes place rapidly, and this method may be used in difficult cases. It is possible that the alcohol may precipitate some of the products of the action of the acid on the material.

RESULTS.

The method is much shorter than the official method. The writer has weighed out a set of samples, digested, filtered, and washed the crude fiber in three hours. Concordant results are more easily obtained than by the other method. Some of the determinations made are as follows:

TABLE II—*Determination of crude fiber by König's method.*

	Per cent.		Per cent.
Timothy hay	27.40	Timothy hay, No. 2	31.95
	27.65		31.60
	27.32		32.04
Corn bran	10.09	Sheep dung	34.33
	9.80		33.94
	10.19		
	10.41		

The work of König and others has shown that the crude fiber is practically free from pentosans.

In the case of cotton-seed meal great difficulty was experienced in filtration, and the crude fiber obtained was much too high. It seems probable that this substance must be subjected to some preliminary treatment before the method can be applied.

This method was given to a graduate student, with the request that he determine crude fiber in two samples. His determinations were 3.5 to 4.9 per cent lower than those of the writer, and the explanation lies in the fact that the glycerol he used was a little more concentrated, and he boiled the mixture more vigorously.

It is believed that determinations made by different analysts with the König method will compare favorably with those made by the official method, especially when chemists have become to some extent familiar with the new method.

CONCLUSION.

The König method has the following advantages:

(1) It yields a fiber practically free from pentosans.

(2) It requires fewer manipulations and less time than the official method.

On the other hand, it requires to be studied in its application to cotton-seed meal.

The writer desires respectfully to suggest to the referee on foods that the study of the König method of determining crude fiber be undertaken.

The President. If there is no objection this subject will be referred to committee B on recommendations, of which Mr. Winton is chairman. Is there anything further in relation to the analyses of foods and feeding stuffs? If not we will listen to the report on ash.

REPORT ON ASH.

By G. S. Fraps, *Referee.*

The referee of the present year continued the work on ash along the line adopted by Mr. Shuttleworth, the referee last year, namely, the study of the method of preparing the ash. The following circular letter was sent out to the chemists of all the agricultural experiment stations in the country:

Dear Sir: The referee on ash of the Association of Official Agricultural Chemists this year will continue the study of the method of preparing the ash. The preparation of an ash that contains all the inorganic constituents lies at the foundation of the ash analysis; it would be worse than useless to analyze an ash from which unknown amounts of chlorin, sulphur, potash, soda, etc., have been volatilized. The point to which special attention will be given this year is, whether any loss of sulphur or potash occurs when the substance is incinerated with calcium acetate in an open dish.

We would like to have your cooperation in this work. Please inform the referee if you will undertake it.

Yours, very truly,

G. S. Fraps, *Referee.*
E. W. Magruder, *Associate Referee.*

Sixteen chemists requested samples and undertook to perform the work if time permitted. Encouraging letters were received from others who were unable to cooperate. W. P. Headden called attention to some work of his relative to loss in sulphur and chlorin in preparing an ash.[a]

Two samples were prepared—of wheat bran and cotton-seed meal. Calcium acetate was prepared by dissolving chemically pure calcium carbonate in chemically pure acetic acid and evaporating the solution to dryness. The following directions for work were sent along with the samples:

DIRECTIONS FOR WORK.

Mix each sample thoroughly and determine moisture in the usual way. Determine potash and sulphur in duplicate in both samples by the methods described below.

Calcium acetate.—Dissolve the contents of the bottle in water and dilute to 200 cc in a graduated flask; 100 cc will contain calcium acetate approximately equal to 1 gram CaO.

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[a] Colorado Experiment Station. Bul. No. 35, 1896.

Sulphur and potash.—Place 15 grams material in a weighed platinum dish and moisten it with 40 cc of the calcium acetate solution. Mix well, dry on a water bath, and ignite to an ash at as low a temperature as possible. Weigh. Dissolve the ash in dilute hydrochloric acid, evaporate to dryness and dry the residue with a little hydrochloric acid, take up with about 50 cc of boiling water, filter, and wash and determine the sulphur by the official method, page 78, Methods Association of Official Agricultural Chemists (1899).

Heat the filtrate, add sulphuric acid to precipitate the barium, and add ammonia and ammonium oxalate. Filter, wash with hot water, and determine potash in the entire filtrate as directed in (3a), page 22, Methods Association of Official Agricultural Chemists.

Potash.—Ignite 15 grams material with sulphuric acid as directed in (b), page 22. Add ammonia and ammonium oxalate to the hot solution, filter, wash with hot water, and proceed to determine potash in the entire filtrate as directed in (3a), page 22.

Please report results as soon as possible, not later than October 1. State weight of material taken, weight of ash obtained by ignition with calcium acetate, weight of precipitates obtained, and percentage of K_2O and SO_3 in the wheat bran and cotton-seed meal.

MOISTURE.

The results on moisture are stated as a matter of record. The fact that moisture determinations may vary considerably has already been emphasized by the referee on foods.

Table I.—*Moisture determinations.*

Analyst.	Wheat bran.	Cotton-seed meal.
	Per cent.	*Per cent.*
C. H. Jones, Vermont	8.93	4.82
E. G. Runyan, Washington, D. C	9.16	4.82
T. M. Price, Maryland [a]	10.18	5.48
F. S. Shiver, South Carolina [b]	10.43	5.62
R. W. Thatcher, Washington (State) [c]	11.18	6.42

[a] Dried sample to constant weight at 100° C.

[b] Dried sample to constant weight in water oven.

[c] Dried sample in hydrogen at the temperature of boiling water.

ASH.

The quantity of ash obtained from 15 grams of material, as received, is given in the following table. The ash contains calcium oxid and carbonate derived from the calcium acetate. Its quantity gives a clue as to the temperature used in ignition.

Table II.—*Ash.*

Analyst.	Wheat bran.	Cotton-seed meal.
	Grams.	*Grams.*
C. H. Jones, Vermont	1.1754	1.5036
E. G. Runyan, Washington, D. C	1.2503	1.5377
T. M. Price, Maryland	1.1754	1.4729
T. S. Shiver, South Carolina	1.2817	1.6172
R. W. Thatcher, Washington (State)	1.2243	1.5100
A. W. Blair, Florida	1.1828	1.4826

REMARKS OF ANALYSTS.

C. H. Jones.—The weight of ash reported contains the added lime and considerable carbon which I could not get rid of by heating.

T. S. Shiver.—These determinations of ash may be a little high, since a small quantity of carbon, especially in the case of the cotton-seed meal, was still left in the same. I have never found simple ignition satisfactory in the determination of ash, and therefore it is my invariable practice to first char the sample, and then extract with hot water. The aqueous extract is evaporated to dryness, gently ignited, and weighed. The insoluble residue is ignited to redness until all carbon is destroyed, and also weighed. By these means I get an ash entirely free from carbon, and also avoid any loss of volatile constituents. In the weights of ash given for the cotton-seed meal and the wheat bran, I have not made corrections for the amount of lime contained in the calcium acetate used.

R. W. Thatcher.—Forty cubic centimeters of calcium acetate solution ignited alone gave 0.6782 gram residue. Correcting for this addition the percentage of ash is:

	Per cent.
Cotton-seed meal	5.51
Wheat bran	3.64

E. G. Runyan.—Ash was determined in the usual way, using 3 grams:

	Per cent.
Cotton-seed meal	6.87
Wheat bran	4.75

The remainder of my report falls naturally into two sections; namely, the determination of sulphur and the determination of potash.

DETERMINATION OF SULPHUR.

The results obtained for sulphur according to the methods already described are given in the following table.

TABLE III.—*Sulphur (SO_3) by calcium acetate method.*

Analyst.	Bran.		Cotton-seed meal.	
	Grams taken.	Per cent.	Grams taken.	Per cent.
C. H. Jones, Vermont	3.7500	0.19	3.7500	0.60
E. G. Runyan, Washington, D. C	1.5000	.18	1.5000	.30
T. M. Price, Maryland	6.0000	.18	6.0000	.36
T. S. Shiver, South Carolina	15.0023	.21	15.0061	.39
R. W. Thatcher, Washington (State)	15.0000	.19	15.0760	.37
G. S. Fraps, North Carolina	15.0000	.22	15.0000	.34
A. W. Blair, Florida	15.0000	.18	15.0000	.36

Mr. E. G. Runyan gives determinations of sulphur in the ash prepared in the usual way, as follows: Wheat bran, 0.015 per cent SO_3; cotton-seed meal, 0.026 per cent. Ignition with calcium acetate therefore gives over ten times as much sulphur in each case as when the material is ignited alone.

VALUE OF THE METHOD.

The results obtained by different analysts with the calcium acetate method agree to a certain extent, and from this standpoint the method could be considered as fairly satisfactory.

Study of the method in another way, however, warrants the assertion that the calcium acetate is valueless so far as the determination of sulphur is concerned. This conclusion is reached after comparing the results obtained by the calcium acetate method with those obtained by the use of another method.

A method was sought by which we could be reasonably certain that all the sulphur contained in plant materials, alone or in organic combinations, would be retained and determined. A great difficulty is that a very small amount of sulphur is contained in a large quantity of plant materials. Several methods were given a trial and finally the one described below was adopted.

NITRIC-ACID METHOD FOR SULPHUR.

Place 15 grams of material in a flat porcelain evaporating dish of about 250 cc capacity, add 35 cc of nitric acid (concentrated), and heat gently until the action has moderated; add 1 gram potassium nitrate and evaporate to a thin paste. Transfer to a platinum dish, evaporate on a water bath, and ignite to an ash. Heat for some time, remove to a porcelain dish and dissolve the ash in dilute hydrochloric acid, evaporate to dryness, and dry the residue thoroughly to render silica insoluble. Moisten with a little dilute hydrochloric acid, evaporate, and dry again. Moisten the residue with about 5 cc of hydrochloric acid, take up with about 50 cc of boiling water, filter, and wash, and determine the sulphur by the official method. (Methods Association of Official Agricultural Chemists, 1899, page 78.)

Make a blank determination, using the same quantities of potassium nitrate and nitric acid, and evaporating off the acid before precipitating.

This method was sent out to a few analysts. The results obtained are as follows:

TABLE IV.—*Determination of Sulphur as SO_3.*

WHEAT BRAN.

Analyst.	In ash.	In calcium acetate ash.	Nitric-acid method.
	Per cent.	*Per cent.*	*Per cent.*
C. H. Jones, Vermont		0.191	0.311
A. W. Blair, Florida		.173	.396
Do		.182	.393
G. S. Fraps, North Carolina		.221	.398
Do			.336
Do			.368
Do			.373
Do			.369
Average		.194	.368
COTTON-SEED MEAL.			
C. H. Jones, Vermont		0.597	0.964
A. W. Blair, Florida		.366	.965
Do		.342	.981
Do		.367	[a]1.09
G. S. Fraps, North Carolina		.343	.928
Do		.328	.930
Do			.941
Average		.390	.951
VARIOUS FEEDING STUFFS.			
Green rape (G. S. Fraps)	1.02	1.25	1.63
Wheat bran (G. S. Fraps)	.00	.14	.30
Corn silage (G. S. Fraps)	.20	.24	.43
Timothy hay (G. S. Fraps)	.15	.17	.28

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[a] This was the first one worked by this method and was not satisfactory. It was excluded from the average.

CONCLUSIONS.

In considering these figures it must be remembered that 15 grams of material is used, and on this account the sulphur trioxid can be calculated to 0.001 per cent.

In the light of these results the only conclusion in regard to the calcium acetate method is that it does not give correct values for sulphur. It is not believed that the loss of sulphur is due to volatilization of sulphates, but to the escape of organic sulphur compounds, which are not burned or oxidized. Most of the sulphur in a plant is not in the form of sulphates, but in an organic form, and it is not surprising that this is lost under such conditions. The same would be true of chlorin, which is probably much more difficult to retain than sulphur.

The nitric acid method gives fairly satisfactory results in the hands of different analysts, and taken in connection with the following work these results, in my opinion, justify its adoption by the association.

MODIFICATION OF THE NITRIC ACID METHOD.

The nitric acid method has been modified in two ways as follows:

(1) Five grams of material was placed in a 3½-inch porcelain evaporating dish, 20 cc of nitric acid (concentrated) added, and the mixture heated cautiously on a water bath until all danger of overflowing had passed. It was then evaporated partly, 10 cc of a 5 per cent solution of potassium nitrate added, the mixture evaporated to complete dryness, and ignited, at first gently, then under a blast lamp, until the residue was white. It was then dissolved in hydrochloric acid, evaporated to dryness, and heated for some time in an air bath, to render silica insoluble. The residue was taken up in water with the addition of a little acid, filtered, and the sulphuric acid precipitated with barium chlorid, etc., in the usual way.

(2) The method is the same as the preceding, save that 5 grams of a mixture of sodium carbonate and potassium nitrate (5:3) is used in place of the potassium nitrate. In this case the ignition must be begun very cautiously, and even with care it is difficult to prevent part of the material being thrown from the dish.

The following are the results:

TABLE V.—*Determination of SO_3 by nitric acid method.*

	Method 1.	Method 2.
	Per cent.	*Per cent.*
Cotton-seed meal, No. 1	1.22	1.12
	....	1.15
Cotton-seed meal, No. 2	1.17	1.05
	1.15	.99
Cotton-seed meal, No. 3	1.33	1.08
	1.28	1.08

The agreement between the duplicates was not as satisfactory as might be desired. It is seen that, for some reason, the first method gives higher results than the second, and has the further advantage that the ignition can be performed more readily.

DETERMINATION OF POTASH.

The results obtained by the different analysts for potash by the methods already described are as follows:

TABLE VI.—*Potash.*

Analyst.	Bran.		Cotton-seed meal.	
	Calcium acetate method.	Sulphuric acid method.	Calcium acetate method.	Sulphuric acid method.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
C. J. Jones, Vermont	1.21	....	2.03	....
E. G. Runyan, Washington, D. C	1.08	1.29	1.76	1.86
T. M. Price, Maryland	1.05	1.21	1.32	1.66
T. S. Shiver, South Carolina	1.26	1.30	1.90	1.94
R. W. Thatcher, Washington (State)	1.22	1.22	1.73	1.98
G. S. Fraps, North Carolina	1.23	1.27	1.79	1.93
A. W. Blair, Florida	1.25	....	1.86	....
Average	1.18	1.26	1.78	1.87

A consideration of these results leads to the conclusion that the calcium acetate method as tested—that is, when the substance is moistened with calcium acetate solution and burned in an open dish—gives results which are too low for potash. These results are not entirely unexpected. Dr. Shuttleworth, who devised this method, advises the use of a closed dish, and states that the ash prepared in an open dish contains less potash than that prepared in a closed dish.

Whether the same quantity of potash can be obtained from a plant when ignited with calcium acetate in a closed vessel as when ignited with sulphuric acid is a question that can be answered decisively only after careful experimental study. The following work by Mr. Hartwell, of the Rhode Island Station, is evidence that the calcium acetate method may not give correct results. This summary is prepared with his permission from a letter to me by Mr. Wheeler.

Analyses were made of the ash obtained from golden millet, obtained in two ways:

(1) The ash was prepared in a closed apparatus (Tucker's) with the use of 50 cc of a solution of calcium acetate, containing 0.5142 gram calcium oxid.

(2) The ash was prepared by charring the material in an open dish and extracting with water, after which the residue was incinerated.

In the first set of experiments, 0.15 per cent less potash was obtained by the use of a closed vessel and calcium acetate than by the method of charring and extracting, and similar losses of sodium and calcium appeared.

In the second set of experiments, ash was prepared by a third method also, namely:

(3) The material was charred in an open dish, extracted, and the residue incinerated with the addition of calcium acetate.

The results were as follows:

TABLE VII.—*Determination of potash as K_2O in golden millet.* (*Hartwell*).

	I.	II.
	Per cent.	*Per cent.*
(1) Calcium acetate in closed dish	1.036	1.027
(2) Charring and extracting	1.055	1.051
(3) Charring and incineration with calcium acetate	1.085	1.071

From this it would appear that there may be some advantage in the use of calcium acetate in the final incineration after the material has been extracted with water.

Perhaps by the use of a little hydrochloric acid during the extraction this would not have been the case. The calcium acetate method, even with the use of a closed dish, gives results too low. Apparently double silicates were produced, and the bases contained in these were insoluble in hydrochloric acid.

CONCLUSIONS.

In the light of this work the only possible conclusion in regard to the calcium acetate method is that it does not give correct results for potash when an open dish is used, and that even when a closed dish is used the evidence is that it may not give correct results.

RECOMMENDATIONS.

(1) It is recommended that the title of this section be changed from "Methods for the analysis of ashes" to "Methods for the determination of inorganic plant constituents."

The reason for making this recommendation is that there is no method known, at least at present, by which a plant can be burned and all of the sulphur, chlorin, and other constituents be left in the ash; that is to say, we are not able to prepare an ash which contains all the inorganic constituents of the plant.

(2) It is recommended that the method for the determination of sulphur in plants called the nitric-acid method (as modified) be adopted as a provisional method. (See (1) modification of the nitric-acid method.)

(3) It is recommended that the determination of chlorin be omitted from the methods until a method can be adopted which will approximate a correct result.

(4) It is recommended that the determination of potash by ignition of the plant substance with sulphuric acid, as described in the determination of potash in fertilizers, be adopted as an alternative method.

I have rewritten the methods of analysis relating to ashes, introducing the recommendations made above and the changes made last year, namely, "the use of calcium acetate and burning ash in some way to prevent volatilization," with the necessary minor changes. The methods are here offered as a part of this report, and without making any recommendation I submit them to the committee, to be dealt with as it deems best.

Methods of Analysis.

VII.—METHODS FOR THE DETERMINATION OF INORGANIC PLANT CONSTITUENTS.

1. PREPARATION OF SAMPLE.

The material must be thoroughly cleaned from all foreign matter, especially from adhering soil. It is to be ground and preserved in carefully stopped bottles.

2. DETERMINATION OF CARBON-FREE ASH.

(a) *Preparation of calcium acetate.*

Dissolve 20 grams c. p. calcium carbonate in c. p. acetic acid, and dilute to a liter. Evaporate 20 cc in a platinum dish, ignite gently, then strongly, to constant weight. The dish must be weighed quickly. This gives the calcium oxid in 20 cc.

An alternative method is to dissolve marble in hydrochloric acid, evaporate, and dry to render silica insoluble, dissolve with water and a little acid, and precipitate iron and aluminum in the usual way. The calcium is then precipitated with ammonia and ammonium oxalate in hot solution, the precipitate washed well, dried, ignited, and weighed. It is then dissolved, and diluted so that 100 cc contains 1.1 grams calcium oxid.

It is best to test the purity of this reagent by making blank determinations with it.

(b) *Preparation of ash.*

Moisten 10 to 20 grams substance with 40 cc calcium acetate, dry on a water bath, and ignite, gently at first, then more vigorously. The quantity of calcium acetate used should be sufficient to prevent fusion of the ash. Some form of apparatus must be used to prevent volatilization, either Shuttleworth's[a] or Tucker's,[b] or an ordinary platinum dish may be used, fitted with a cover, like that described by Wislicenus.[c] The weight of the ash must be corrected for lime, carbon dioxid, and carbon.

(c) *Determination of carbon dioxid.*

Using the ash prepared in (b), liberate the carbon dioxid with hydrochloric acid in any of the usual forms of apparatus, determining the carbon dixoid evolved either by increase of weight of potash bulbs or loss of weight of the apparatus. The former method is preferred.

(d) *Determination of carbon, sand, and silica.*

The residue from the carbon dioxid determination is transferred to a beaker or evaporating dish, evaporated to dryness, and thoroughly dried and pulverized to render silica insoluble. The dry residue, etc. (See Bull. 46, Rev. Ed., Div. of Chem., U. S. Dept. Agr., p. 78, and end of section 2.)

Subtract carbon, carbon dioxid, and calcium oxid added in the form of calcium acetate from the ash, and calculate results as carbon-free ash.

3. DETERMINATION OF MANGANESE, CALCIUM, AND MAGNESIUM.

As on page 78 of the "Methods," unchanged, save that the word "ash" is inserted after "grams," line 2; add "The quantity of calcium found must be corrected for the calcium added."

4. DETERMINATION OF PHOSPHORIC ACID.

(a) An aliquot portion of the hydrochloric-acid solution, corresponding to 0.2 to 1 gram ash, is to be used for the determination by any of the methods described for total phosphoric acid in fertilizers.

(b) The determination can also be made in the plant substance as in (a_2), page 12 of the "Methods" for phosphoric acid, using sufficient material to give from 0.2 to 1 gram ash in the aliquot portion of the solution taken for the phosphoric acid determination.

5. DETERMINATION OF ALKALIES.

(a) An aliquot portion of the hydrochloric-acid solution (see 2) corresponding to 1.5 to 1 gram of ash is evaporated to dryness. Redissolve the residue in about 50 cc water, add milk of lime or barium-hydroxid solution, which must be perfectly free from alkalies, until no further precipitation is produced.

(b) Potash may be determined as directed for potash in organic compounds (b), page 22 of the "Methods," using sufficient plant material to get from 0.5 to 1 gram ash in the aliquot portion of the solution taken for the potash determination.

6. DETERMINATION OF SULPHUR (PROVISIONAL METHOD).

See recommendation No. 2 above, and (1) under "Modification of the nitric-acid method."

The PRESIDENT. Is there any discussion?

Mr. WHEELER. I would like to ask the referee a question, if I may. I would like to know if he has tried the method of burning in a glass tube?

Mr. FRAPS. I have not tried that. The materials that we have been working upon usually have a large quantity of organic matter, and I think it would give a little trouble.

Mr. CAMERON. Has the referee used the method of Carius?

Mr. FRAPS. The method of Carius I have never used with my plan. We constantly find a large quantity of organic matter present, and I think there would be a high pressure inside of the tube, which would make it very liable to explode unless you relieved the pressure by opening the tube. Further, we have none of the special tubes which must be used for this purpose.

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[a] Exp. Station Record **11**, 304.

[b] Ibid., 506.

[c] Zeitschr. f. anal. Chem., 1901, **40**, 491.

Mr. Huston. How about the method of determining sulphur in coal? Have you tried the Eschka method?

Mr. Fraps. No; I have not tried it.

Mr. Huston. It seems to be successful in determining sulphur in coal.

Mr. Fraps. The trouble is the stuff is so volatile.

Mr. Huston. I do not think it would be any more volatile than bituminous coal.

Mr. Wheeler. We have had some difficulty. We do not know how to make the mixture of all substances in which we must determine ash. There are some materials that give no difficulty, and there are others that are extremely difficult. With chicory we could only obtain correct results for moisture by drying in a vacuum over sulphuric acid. But before we get down to the solution of this matter we must take up the question of how to determine the moisture in various kinds of material, and our methods of procedure must be adjusted to each kind of material that we are going to analyze. I am sorry to throw cold water on the matter in this way, but I feel that there is no general rule that can be applied to all organic substances.

Mr. Fraps. I would like to state that these methods represent the best experience we have at present, and for that reason it seems advisable to use them until we can devise methods adapted to other materials and improve upon them.

The President. Is the referee on tannin present?

Mr. Barnard. I was told that the referee on tannin would not be present until Saturday.

The President. There is one matter that might be brought up now, viz, the fixing of the time at which the committee on nominations shall make their report.

Mr. Wiley. I move that it be 3 o'clock on Friday—a special order.

The President. The motion has been made and seconded that the report of the committee on nominations be made the special order of business for 3 o'clock on Friday. It is adopted.

Mr. Wheeler. Are matters of general business in order?

The President. What is the nature of it?

Mr. Wheeler. A suggestion has been made, in view of the large number of referees and the great number of questions that are to be acted upon if possible, that the committee on recommendations of referees for next year be appointed at this meeting. I would suggest that if this general plan is adopted we begin next year on that basis. We have the committees already appointed for this meeting, and the recommendations made by the referees should be referred to the committee for report next year, and in this way every member of the association would have a printed list of all matters that are to be acted upon placed in his hands before the end of the year, and the committee would have the whole year in which to consider the recommendations. As it is now it is very difficult for the committee on recommendations to act on the report in a satisfactory manner at the time of the meeting, while formerly it was comparatively easy. It is thought that perhaps some scheme of this kind would be desirable. Of course it would not be put into operation this year, because we have our committees already appointed, but this is brought forward now with the idea of adopting it for the future. It would not involve any change in the constitution.

The President. The simple action of the association would be sufficient.

Mr. Ross. I think it is a very good suggestion to have the subjects referred to the committee, of which I happen to be a member, in advance. I know I could report to very much better advantage if I had a year in which to study them. It is a very good suggestion.

Mr. Wheeler. I move that the committees on recommendations of referees for next year be appointed at this meeting so that these committees may have time to consider the recommendations. The idea is that the recommendations will go to the committee and they will have their final recommendations printed and sent out by the committees for consideration.

The President. You mean that this shall take place next year?

Mr. Wheeler. It can not apply this year, but at next year's meeting. If all the recommendations go to this committee they will act upon them as soon as possible, and after being acted upon have copies printed and sent to the members of the association, so that when we come here we shall all of us have been able to consider the details of the propositions recommended by these committees, and in that way we can all act more intelligently.

The President. In that way there would be no reports on recommendations?

Mr. Wheeler. Not next year, under that system.

Mr. Wiley. Would it not be well in appointing a committee for next year at this session to instruct the referees to furnish the chairman and members of each committee with copies of his recommendations at least a month before the meeting of the association, so that the committee would have fully considered the recommendations before the association meets?

The President. The idea was to have no report next year from the committee on recommendations of referees.

Mr. Wiley. That plan might postpone some action which would be very important, whereas it seems to me that if the referees be instructed, as I have just mentioned, to furnish the committees now to be appointed with copies of their recommendations at least a month before the meeting of the association, fully matured discussion of it could be had by the committee. At least each member could separately study these recommendations and be ready to act intelligently and without being pressed for time.

Mr. Wheeler. I am most heartily in favor of that, whether the report be made next year or a year later, but I doubt very much if it would be possible for the referees to get their reports in so that they could furnish this report to the committee. A number of referees have difficulty in getting their reports in time to write up their papers, and some have been reported after they have arrived at this meeting. I fear that can not be done.

Mr. Huston. It seems to me that we might make a compromise between the present method and that suggested. Sometimes there is a necessity for immediate action. Very often there is no necessity for any delay. I have been on that committee for a good many years, and part of the work can be solved very quickly, while there are some questions which require time and care to answer, but I believe that the committee should be appointed this year, if necessary, to report such things as they see fit at the next meeting. Those things which are necessary can be attended to at this meeting, but those things which are doubtful should be more carefully considered. The proposed compromise between our present method and the one suggested would be a good working solution of the problem.

The President. As there is no motion before us, the chair would suggest that a motion be made appointing a committee to put this affair in shape and then report later. A motion is in order to appoint such a committee if you so desire.

Mr. Huston. I hardly think it is necessary to appoint such a committee. If, during the present year, the committee on recommendations, any of the fifteen members you have appointed, should find some work upon which they desire more time, all they need do is to ask that they be granted more time, or that the matter be referred to the committee for the following year. This can come in with the report of the present committee and will save considerable time.

Mr. Wiley. I would like to say that if the chairmen of the committees will take these reports which the referees have handed in with them to-night, they would be able to begin at once on the work that has already been presented. The stenographer will give the reports to the committees.

Mr. Huston. All the reports so far go to Committee A.

Mr. Wiley at this time explained to the convention that a photographer desired to take a picture of the members the following day, and suggested that this be done at noon, which was agreed to.

The convention then adjourned, to meet the following morning at 9.30 o'clock.

MEMORIAL EXERCISES IN HONOR OF THE LATE JOHN A. MYERS.

The Washington section of the American Chemical Society invited the Association of Official Agricultural Chemists to a joint meeting in the hall of the Cosmos Club on Thursday evening, November 14. After the meeting of the chemical society was adjourned, Mr. Van Slyke took the chair and stated the object of the after meeting to be memorial exercises in honor of the late John A. Myers, one of the founders of the Association of Official Agricultural Chemists. Mr. Van Slyke spoke as follows:

We have met at this time to do honor to the memory of one of our members who had been actively identified with our association from its very beginning, who was with us one year ago in the full vigor of a splendid manhood, but who since then has been called from his earthly labors—I refer to Mr. John A. Myers. We all remember Mr. Myers for his ever genial, happy, cheerful spirit. To know him was to appreciate his sunny, hopeful disposition and to desire his friendship.

Mr. Myers was one of the charter members of our association and was president in 1889. He was seldom absent from our annual conventions and was usually active in their proceedings. His presence will be missed by those of us who knew him at all well. There is, therefore, peculiar appropriateness in holding a special session of our association to do honor to the memory of him who was so well known and generally esteemed.

Our secretary, Mr. Wiley, has prepared an address on the life and public services of Mr. Myers and will now present it.

REMARKS OF MR. H. W. WILEY.

Mr. President, members of the Washington section of the American Chemical Society, and members of the Association of Official Agricultural Chemists: It was my good fortune to have known Mr. Myers from the time we were both students until his death. I first met him in the laboratory of the Berlin University. I remember well his work there, his faithful service, and his universal popularity among the students. He displayed, even at this early date, those qualities of heart and head which continued to increase with his growth and which commended him so favorably to all those with whom he was brought in contact. I think I know something of Mr. Myers's characteristics, his strong and weak points, for the best of us can not claim exemption from imperfections.

The most prominent characteristic of his personality was his absolute sincerity. In his intercourse with his fellow-students he never had anything to conceal, nor did he ever try to present himself in a false light. After his return to the United States I kept in constant touch with his career, with his services at Butler College, Indianapolis, in the Mississippi Agricultural College, as director of the Agricultural Experiment Station of Virginia, and later in his last capacity as director of the nitrate of soda propaganda in the United States. That part of his career which we remember most vividly, perhaps, is his connection with our association. He was present as one of its founders, and of the seventeen meetings of the association held during his lifetime he attended twelve. In this respect his example is commended to many of our directors of agricultural experiment stations who in the push of executive work sometimes seem to lose their interest in their old profession or have no time to manifest it. We congratulate our science because it has furnished so large a percentage of directors of agricultural experiment stations. Almost 50 per cent of the directors in this country and more than 70 per cent in Europe have been professional chemists. We are glad to contribute of our force to the work of direction, which we regard as of a higher character even than that of analytical practice and almost as high as that of research, but in making this contribution we hope that the interest in our own affairs will not be lost and that we shall still have the cooperation and aid of those who were formerly associated with us.

In the social affairs connected with our meetings Mr. Myers always took an important part. His good nature, his jovial appearance, and his hearty laugh were always welcome and were always missed when absent. There was no one more welcome at a dinner and no one enjoyed to a greater extent the wit and wisdom of the after-dinner speeches. After all there is nothing in life quite equal to association with a true friend. "Nil ego contulerim jucundo sanus amico," said Horace truly two thousand years ago, and the worth of this sentiment has only increased with the passing centuries.

During the last few years of his life Mr. Myers entered a new field of activity, which brought him into more intimate contact with the agricultural experiment work of this and other countries. As the representative of the syndicate controlling the output of nitrate of soda of South America, he founded in this country a propaganda unique in its scientific relations. Mr. Myers, in his capacity as the representative of this syndicate had nothing whatever to do with the sale of this useful plant food. He simply studied in a general way the needs of the great staple crops of the country for available nitrogen, and collected data and disseminated scientific articles in relation thereto. It was while engaged in this work in California and in preparing for a visit to the Hawaiian Islands that he contracted typhoid fever, which proved fatal. Although far from his home, friends were not wanting to minister to his wants, and it is a consolation to his family that nothing was left undone, either on the part of the hospital authorities or his friends, to make his days as comfortable as the disease would permit. Fortunately his wife was able to reach his bedside two days before his death, while he was still in full possession of all his mental faculties.

When it was proposed to the executive committee that some formal memorial exercises be held at the present meeting, they unanimously approved of the plan. The invitation of the American Chemical Society to join in such a meeting was forwarded to each member of the executive committee, and the following replies were received:

Permit me to state that I am thoroughly in accord with the idea that we should accept the invitation so cordially extended by the Washington section of the American Chemical Society. It is with peculiar pleasure also that I note the proposed meeting for the purpose of paying a tribute to the late John A. Myers. I sincerely feel that every member of our association will be most heartily in accord with such a plan.

Very sincerely, yours,
H. J. WHEELER.

A memorial meeting in honor of Dr. Myers will be a very proper and desirable act on the part of the Association of Official Agricultural Chemists.

Very truly, yours,
L. L. VAN SLYKE.

It meets entirely with my approval to accept the invitation of the Washington section of the American Chemical Society for our association to meet with them in the hall of the Cosmos Club on the evening of November 14, and later to hold a session of the Association of Official Agricultural Chemists in the same hall for the purpose of paying a tribute to the late Dr. John A. Myers. I am especially glad that the latter meeting has been arranged, as I remember Dr. Myers most pleasantly, both as a professor in our institution and in our frequent meetings in later years. I regret my inability to be present at the meetings.

Yours, very truly, W. R. PERKINS.

I am heartily in favor of the joint meeting of the Association of Official Agricultural Chemists with the American Chemical Society, and only regret that I shall be unable to attend this year.

Sincerely, yours, F. W. TRAPHAGEN.

I wrote to the Permanent Nitrate Committee in London, informing them that this memorial meeting would be held, and asked for some expression in regard to the exercises. In reply to my communication I received the following letter from the secretary of the committee:

PERMANENT NITRATE COMMITTEE,
3 GRACECHURCH STREET, E. C.,
London, November 2, 1901.

DEAR SIR: My committee tender to you their cordial thanks for the opportunity you have afforded them to place on record their appreciation of the work and character of the late Dr. John A. Myers. I would say that at the meeting of my committee next after the receipt of the sad news of Dr. Myers's decease their first act was to record their sense of the great loss which they had sustained in one whose ability, energy, and devoted services during the previous three years they had so highly esteemed.

Thorough in everything he did, he died doing his duty with all his strength.

I am, dear sir, yours, faithfully,

JOSEPH HILLMAN, *Secretary*.

Unusual attention naturally attaches to the last days of a friend's life, and so the following account of the last work done by Mr. Myers will be especially grateful to his friends. This memoir was written by Mr. Charles H. Shinn, of the College of Agriculture of the University of California.

BRIEF RECOLLECTIONS OF THE LATE JOHN A. MYERS.

By CHARLES H. SHINN.

I take it for granted that Dr. Myers in his own correspondence covered the ground of these brief notes. But he was, of course, writing from his own standpoint; and as no man realizes the impression he makes upon others, the standpoint of the writer may be of some interest.

It goes without saying that the personality of Dr. Myers was to all who met him extremely pleasant. He was a man who grew upon acquaintance. He kept to a rare degree the "boyishness" which most men lose, and he was extremely sensitive to praise or blame, very high minded, and much in earnest in all he undertook. He put the interests of his business first and foremost; worked early and late to get through with his correspondence and to push business. His pleasures were simple and refined. I am constantly hearing from people who met him during our trips in California, all regretting his loss.

We left San Francisco in February, 1900, and went to the Cloverdale Citrus Fair. I had been invited to deliver an address there and incidentally to judge the fruit. I wrote the committee that if they so desired I could bring Dr. Myers with me, and they were very glad to have him come. We were met by the citizens and shown every attention. When it came to the special work of the occasion we found the hall extremely large, and very noisy. We both felt that little good would come of the effort to speak there. I asked to have Dr. Myers introduced first, and although he had said he knew "nothing about oranges," it turned out that he knew a great deal about his subject. He gathered up sample oranges from the various exhibits and took them forward to the edge of the high platform (which was really an outjut from the balcony), and, cutting them apart, talked to the audience about "good and bad oranges," etc. He was clearly heard over most of the barn-like building, and the local newspapers spoke very nicely of his effort. I understand that a good deal was printed, and I shall try to get copies.

When it came to the judging of the fruits the local committees backed out entirely. We were forced to spend about fifteen hours on two successive days in cutting open and testing fruits according to the latest California scale. This is a modification of the American pomological scale for citrus fruits. Dr. Myers showed the greatest patience and industry, going over and over the various tests with me and checking off results, suggesting modifications, and, in short, rising to the occasion with astonishing good nature. The whole work was really thrown upon us, as the people in this district had never before had their fruit judged according to official standards.

We were together every evening and met the people of this little country district. Dr. Myers told stories, made himself entertaining to men, women, and children, and won golden opinions. He was not well, but no one would have known it from anything that he said, except that once or twice he told me that "he did not feel in good form." He was very much interested in the Sonoma Valley and its many beautiful places. He even said he would "like to bring his wife and daughter to see California again some springtime." He talked a good deal about his home and the happiness of his life. He told little stories of his boyhood. He told me about his father, his own education abroad, and a good deal that was interesting and human. We received a cordial invitation to come again next year and a special invitation was extended to Dr. Myers.

Dr. Myers then planned a trip to Oroville and the orange region in the interest of his work, and he was kind enough to say that if I would go with him he would accomplish a good deal more. We talked of it to some degree, but I found it difficult to leave my own university matters, and so I was planning to write as many letters of introduction as possible, and let him make the trip alone later.

Dr. Myers wrote me that he did not feel at all well, and so wanted to go to Paso Robles, take the hot baths, and then go south to Los Angeles, where I could join him. He seems to have had a pleasant visit at Paso Robles substation, where the foreman, Mr. Barber, drove him around the region, and he had a good rest. I then met him in Los Angeles, and we took a trip together to the Pomona substation; went around the town to some extent, and to Santa Monica. From there Dr. Myers went to Santa Ana, and saw the celery growers; then made a second trip to Pomona, and from there to Riverside and Redlands. At the latter place he arranged for some fertilizer experiments, and I believe that he had something under way at Westminster or Santa Ana.

At Pomona Dr. Myers and I dined with Mr. and Mrs. Mills and were driven around the valley. He was in better health than I had seen him for some weeks and was extremely jolly. He told stories all day, and we were trying in vain to cap each story. We called him a "Methodist minister," and told him he "should have a chicken dinner." I never saw Mr. and Mrs. Mills more amused and delighted. They have written me since that no one who ever visited them was better company and Professor Devol, of Redlands, wrote in much the same way.

While in Los Angeles we were out together and spent Sunday afternoon and evening with the family of Will E. Chapin, who is the artist and cartoonist of the Daily Times. When we returned from this visit I took the late train to San Francisco, and Dr. Myers did not go with me to the train, as had been his previous custom, because he said he "was feeling a little tired."

I had letters from him almost daily and I wrote him myself from various places.

Every one of his brief notes on business was cheerful and interesting, but I still got the impression that he was not feeling well, and on several occasions he spoke of friends of his who had passed away and of various other illnesses; then he would brace a little and say this was all nonsense and that when his cold was over he would be all right again.

I noticed with special interest from time to time the ease of his manners with the general public. He was a genuine American of the Middle West, refined by travel and study. He liked people. Naturally people liked him. It was easy for him to get along with everyone he met. Brakemen, conductors, waiters, bootblacks, and the general run of mankind liked to meet him and do things for him. He was never too tired to scatter sunshine as he went.

I had thought that I might be able to be a little more particular in some of these items, but I find that after all I have only the general impression of a thoroughly kindly and real personality mingling in the daily life of all he met. I do believe that none of this is really lost, but that the happiness he gave to his friends and to other acquaintances is a permanent part of their lives. We shall miss him and think of him often, but not without hope, nor cheerlessly. So strongly vitalized a personality, working good in its own way so many years among men, is not destroyed, although it has passed from sight. If there is one lesson among all others which we derive from religion and philosophy it is that we can go forward hoping, not fearing, and that in the hereafter we shall see face to face where now we see but blindly.

Mr. M. A. Scovell contributed a brief sketch of Mr. Myers to the Lexington (Ky.) Leader for April 12, 1901, which was in part as follows:

> The friends of Dr. and Mrs. John A. Myers learn with sadness of the death of Dr. Myers in San Francisco, Cal., April 8, 1901. Only a few weeks ago he left his office in New York in the best of health for California, and now the sad news reaches us of his death, caused by typhoid fever, after an illness of only two weeks.
>
> Dr. Myers is well remembered in Lexington, having been at one time professor of chemistry and physics at Kentucky University. While here he married Miss Minnie B. Plunkett, who survives him. * * *
>
> Dr. Myers was an indefatigable worker in his chosen field of science, and as an investigator he stood among the first. * * * A busy man, always with some important work on hand, he was never so busy but that he could help a friend. His greeting was cordial, his hand shake came from the heart. His personality attracted and held as a magnet all who intimately knew him. Full of life, full of hope, full of ambition, he inspired, he gave cheer, and spread sunlight over all he met.

A lifelong friend and early teacher of Mr. Myers is Prof. A. E. Dolbear, of Tufts College. In answer to my inquiry concerning the early days of Mr. Myers at Bethany College, where Mr. Dolbear was then professor, he wrote me the following:

> As to Myers's college life at Bethany College I remember some things very well. He came without much preparation, but was full of vigor, dead in earnest, knew what he wanted, and was willing to dig for it. At first the other fellows thought he needed discipline, and arranged to haze him. At the end of the mêlée he was not much hurt, but the other fellows needed what bandages, liniment, and court-plaster there was in the town. After that he was let alone. His rank was good in his class work, and he never got tired. In the chemical laboratory he was painstaking, and learned pretty thoroughly what was the meaning of the work he did. At the table he was hearty, and to the dismay of the boarding house would call for more butter and eggs when the plates had been emptied. Such things showed he could be trusted to look after his health.
>
> I do not remember that he took part in any of the college sports, nor do I think he had any of the mild vices. After his graduation at Bethany he took a course of experimental physics at Tufts College, and then went to Europe to study chemistry. He was altogether honorable, and I wish he might have lived to patriarchal age.
>
> I have seen very little of him for the past twenty years, and doubtles you know far better of his work. I should be glad to add to this, but the time I knew him was nearly thirty years ago.

An interesting letter from Gen. Stephen D. Lee, president of the Mississippi Historical Society, and former president of the Agricultural and Mechanical College of Mississippi, gives a short account of Mr. Myers's service in that institution:

> Prof. John A. Myers was elected to the chair of chemistry in the Agricultural and Mechanical College of Mississippi in 1882, and filled the place for eight years. He was a most accomplished chemist, superintended the erection of the chemical building, and instructed all classes to the great satisfaction of the board of trustees and the president. During the time he was at the college several young men graduated in his department with honor, and are now filling prominent positions in several States. Among them may be mentioned Prof. H. H. Harrington, of the Texas Agricultural College, and B. W. Kilgore, director of the North Carolina Experiment Station. Professor Myers was an accomplished gentleman and a most industrious and painstaking worker.

Mr. President, there are other gentlemen here who desire to make some tribute to the memory of Mr. Myers, and I reserve the rest of my remarks until they have been heard.

REMARKS OF MR. B. B. ROSS.

The first intimation I received of the death of Mr. Myers came to me through Mr. Wiley, whom I met in London during the past summer, and it is needless to say that I was greatly shocked at the sad intelligence, as I had anticipated calling on him at his New York office upon my return to this country.

Only a few months before going abroad I had the pleasure of entertaining Mr. Myers at my own home, and at that time I well remember he exhibited in a marked degree those sunny, genial, and amiable traits which were so strikingly characteristic of the man, and to which such happy references have been made in the letters of friends to which you have just listened.

My acquaintance with him did not extend over so long a period as that of some who have already spoken, and dates from the third annual convention of the Association of Official Agricultural Chemists, the first which I had had the opportunity of attending. I remember quite well that, although an almost total stranger in that gathering, I received a most kindly greeting and welcome from Mr. Myers, and when, with some trepidation, I had ventured to take part in the discussion of the association methods, I now recall with pleasure the fact that upon that occasion, as well as at subsequent conventions, his kindly words of encouragement to myself and other young men in attendance did much to make us feel at ease and at home in the meetings of this body.

From that date up to last year I met him at frequent intervals, and as my opportunities for knowing him increased I became more and more impressed with those traits and characteristics which endeared him to his many friends.

This is the first meeting for many years that I have missed his well-known presence in this gathering, and, although he is gone from us, the recollection of his genial nature, his kindly greeting, and his friendly hand clasp will linger with me as a pleasant memory.

REMARKS OF MR. KILGORE.

It was my good fortune to know Mr. Myers well. For eighteen years he was to me one of the best of friends, and I acknowledge publicly now, as I have done frequently in a private way during his lifetime, my indebtedness to him for many favors and kindnesses. As a student in college I came under the influence of his instruction as professor of chemistry in the Mississippi Agricultural College. In the capacities of student, assistant, and guest in his home the different phases of the life of the man in whose memory we have met to-night became familiar matters to me, and I therefore feel specially competent to speak concerning him who has so recently gone from us, leaving great sorrow in the hearts of his many friends, but the most pleasant memories of a useful life, full of service to his fellow-man and to his chosen line of work. I have always regarded Mr. Myers as one of the best of teachers, because he possessed the power and capacity in a marked degree of inspiring and obtaining work from students where others could not get it. It was either work or get out of his classes, and in consequence of this not a few of his students did not regard him pleasantly during their early student days, but by the time they finished their college work and were ready to enter seriously into the realities of life the early feeling of dislike had given place to one of high regard. They now knew the man as he really was, and that, though seemingly hard, he had all along been doing the very best thing for them. He was eminently a success as a teacher, and I have had occasion recently and since his death to speak with some of his former students, who have spoken of him in accord with the remarks I have made.

He did a great service in assisting in the organization and development of the Agricultural College of Mississippi, under the presidency and leadership of that magnificent type of Southern man, Gen. Stephen D. Lee. Mr. Myers was an organizer and leader of undisputed ability, and it was always known where he stood in matters of discipline, policy, and right. Of this work General Lee has spoken, in his communication to the secretary of this association, in unmistakable terms, and of course with authority. This phase of our much-beloved friend's life—that of teacher and college man—I think few, if any of you, are familiar with, and I thought that it would be of interest for me to refer to it here, though I have done so in but a brief and very imperfect way, and I need not say to you, gentlemen, that it brings to me great sorrow.

Mr. Wiley has spoken of Mr. Myers's early connection and earnest and continuous interest in the organization, progress, and growth of the Association of Official Agricultural Chemists to the time of his death. A great deal of this is a personal memory to most of you. I feel specially kindly to him for the help he gave me as a young worker in this association, and for the encouragement received from him and from other older workers, to take the modest part I have in the association work. They have encouraged us to speak out in meeting on all subjects on which we felt we had experience and information that would throw light on the varied and important work that was being done. This spirit of making all feel that they were at home and a necessary part of the work that was being done has helped to make this association what it is, and we love it, and revere and honor the memories of the men who have been its active workers. Mr. Myers's important and valuable work in connection with the experiment-station movement in this country, in which we are all so much interested, has been spoken of, and besides is a matter of memory. For these reasons it is not necessary for me to review this phase of his life work.

We see, though, from the various lines of work to which he has turned his hand with unfailing success, that his life was an exceedingly active one, and he departed it in the midst of great usefulness and in the prime of strong and vigorous manhood, to be mourned by a host of admiring and loyal friends, and most tenderly by a devoted wife and loving daughter. May God bless the broken home and sanctify to the good of all who knew him the memory of his life work.

This, Mr. President, has been a very sad duty for me, and it has been but imperfectly done. To give more appropriate and official expression to our regard and estimate of the services of our departed friend and coworker, I move that a committee be appointed to draft suitable resolutions to be incorporated in the proceedings of this meeting.

This motion being carried, Messrs. Wiley, Kilgore, and Voorhees were named as the committee to draft said resolutions.

REMARKS OF MR. VOORHEES.

I can only echo the sentiments already expressed by my confrères, Messrs. Wiley, Kilgore, and Ross, though I can not let the opportunity go by without giving voice to my deep admiration and love for our departed associate. His was a life that was helpful to all with whom he came in contact. My acquaintance with him began about 1882, when, after his return from Germany, he visited the experiment stations of the East, and the respect formed for him then grew, as my acquaintance ripened, into friendship and love. He was helpful always, and I well remember his words of encouragement and assistance when I first attended the meetings of this association. He it was who first met me and insisted that I should give to the meeting the results of the study of methods which he knew had been carried out at our station, and always at any of our meetings he took pains to bring forward and encourage young men. In later years it was my privilege to come into frequent contact with him, both in a social and a business way, and the more I knew of him the more I admired him for his generous, helpful character. He looked on the bright side of life, was always cheerful and hopeful, and yet forceful. He made things go by sheer force of character. As an association we have lost an able and faithful coworker, and as individuals we have lost a true and tried friend.

Mr. Wiley. Mr. President, I wish to present the following biographical sketch of Mr. Myers:

BIOGRAPHICAL SKETCH OF JOHN A. MYERS.

John Alva Myers, A. M., Ph. D., was born May 29, 1853, on a farm near West Liberty, Ohio County, W. Va., where he resided until after he completed his college course, working upon the farm during his college vacations, and knowing nothing of city life until after his graduation. He prepared for college at the West Virginia State Normal School at West Liberty, and graduated at Bethany College in the A. B. course in June, 1875. During his last year at college he was placed in charge of the chemical laboratory of that institution, which at that time was one of the most complete in the Southern States. After graduating he remained one year in that institution, taking post-graduate studies and teaching analytical chemistry. He was elected professor of chemistry and physics in Butler University, near Indianapolis, Ind., where he organized and fitted up their chemical and physical laboratories. At the end of one year he resigned his position to continue his studies in the universities of Germany, where he remained three and a half years, studying and carrying on original investigations in the chemical laboratories at Göttingen, Breslau, and Berlin, under the immediate guidance of some of the most celebrated professors of Europe. His vacations were used for traveling, which enabled him to travel over nearly all of Europe, through Egypt, Palestine, and portions of Turkey and Greece. During his last year in Germany he was elected professor of chemistry and natural history in the Kentucky University, which he reorganized, and where he added materially to the equipment and efficiency of the chemical laboratory.

From Kentucky he was invited to the Agricultural and Mechanical College of Mississippi. Here he planned, built, organized, and equipped their splendid chemical laboratory, one of the best in the South, and also organized the work of the State chemist, being the first State chemist of Mississippi. He also organized the chemical work of their agricultural experiment station, of which he was the first chemist, and in conjunction with Prof. F. A. Gully, the professor of agriculture in that institution, he organized the first Farmers' Institute ever held in the State of Mississippi. During his stay of seven years in that State he saw the agricultural interests of the State materially advance through these channels.

In 1888 he was invited by the board of regents of the West Virginia University to return to his native State and organize the West Virginia Agricultural Experiment Station. The entire organization of the experiment station, the planning of its buildings, their equipment, the organization of the work, and the assembling of the magnificent collection of apparatus, libraries, etc., were also effected under his direction. He also organized the West Virginia Agricultural College, of which he was dean, and the farmers' institute work in that State, as well as the work of the State chemist of West Virginia. After continuing in this work for ten years, Mr. Myers was appointed manager of the Propaganda for Nitrate of Soda in the United States, since which appointment there have been added to the territory under his charge Cuba, Porto Rico, the Sandwich Islands, and the British Provinces of North America.

Mr. Myers was one of the founders and ex-presidents of the American Association of Agricultural Chemists, which is everywhere recognized for the accuracy of its scientific methods. He was one of the founders of the Association of Agricultural Colleges and Experiment Stations. He served as a member of its executive committee for a number of years, and was also one of its vice-presidents. He has also served on various State boards and was frequently commissioned by the governor of West Virginia as a delegate to National Agricultural Development or Immigration associations.

Mr. Myers was also a member of the Berlin Chemical Society, the American Chemical Society, and Fellow of the American Association for the Advancement of Science.

Mr. Myers's scientific work has been published largely in the form of reports, bulletins, pamphlets and articles in the agricultural press, and was received with such favor that portions of it have been translated for use in Germany and Japan. His work has been preeminently one of organization, in which he was so thorough, energetic and effective that the laboratories left by him have, in several cases, scarcely been changed since he left them.

Mr. Wiley (continuing). Mr. Myers died at Waldeck Hospital, San Francisco, on April 8, 1901, of typhoid fever contracted either on his way to or during his stay in California. His body is buried in the cemetery in Lexington, Ky., and a movement is on foot among his friends to erect a proper monument to his memory.

I can not forbear, Mr. President, on this occasion to call attention to a loss which agricultural science has lately suffered in the death of one of our colleagues in Europe—Prof. Max Maercker, of the University of Halle. Maercker's personality was to me so very much like that of Myers—whole souled, hearty, the best of companions, the most hospitable of hosts, and the most agreeable of guests. Many of us remember his long visit to Chicago during the time of the World's Fair, and I recall with special pleasure my association with him during his visit to Washington and my stay with him at Halle in 1898. I remember him so well when, late in the evening, perhaps almost morning, he stood upon the steps of his residence and called his last farewells to me and his best greetings to his brethren in the United States. His death at the early age of 59 occurred on the 18th of October at Halle. I wished to associate his name with that of Mr. Myers.

The operator of the lantern will now throw upon the screen an excellent portrait of Mr. Myers, taken from one of his latest photographs, which will be reproduced as the frontispiece in the next volume of the proceedings of this association. I will ask that all persons present rise and stand in silence for a few moments in this last official tribute to his memory.

RESOLUTIONS ADOPTED BY THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS IN COMMEMORATION OF THE LATE JOHN A. MYERS.

Whereas the members of this association have learned with profound regret of the death, on April 8, 1901, of John A. Myers, one of its founders, whose services to the association have been faithful and efficient, and whose genial personality was beloved by all his colleagues, be it

Resolved, That we place on record in the files of our proceedings an expression of our appreciation of the aid which his unwavering interest and cooperation have been to us in the past, and the inspiration which his memory will be to us in the future;

Resolved, further, That a copy of these resolutions be transmitted to Mrs. John A. Myers with an expression of our personal association in the grief which she has experienced in his premature death.

H. W. Wiley,
B. W. Kilgore,
E. B. Voorhees,
Committee.

SECOND DAY.

FRIDAY—MORNING SESSION.

President Van Slyke called the meeting to order at 10 a. m.

Mr. Kilgore reported for the committee appointed to wait upon the Secretary and Assistant Secretary of Agriculture as follows:

The committee has performed its duty and found the Secretary out of town, but his secretary promised to give the message to him, and it is likely that he will visit the association. The committee also called upon the Assistant Secretary of Agriculture and extended to him an invitation to attend the proceedings of the convention.

On motion of Dr. Bigelow, the report was approved and the committee discharged.

The PRESIDENT. We will take up now the report on liquor and food adulteration.

REPORT ON LIQUOR AND FOOD ADULTERATION.

By W. D. BIGELOW, *Referee.*

I shall bear in mind the suggestion made by the president in his opening address. It was, in brief, I believe, that the least important portions of the report be omitted in reading. So I shall not read this morning the whole report which the committee has to present. We shall probably save several hours by it. You are probably all aware that in the address of the retiring president, Mr. Kilgore, a year ago, it was suggested that the work of the referee on liquor and food adulteration be increased, or rather that the subject be divided into a number of classes, each of which should be put in charge of one man to prepare methods for the examination of foods. This was acted on, with the result that fifteen classes of foods were arranged and associate referees appointed for each one, or, rather, each one put under the direction of one member of the association. In some cases one man was given more than one class. Unfortunately it happened, after the meeting had adjourned and the men were notified and asked to cooperate with us, that several were unable to do so, and it was necessary to make changes. This was very unfortunate because of the fact that some on whom we had counted for excellent work, who were fully qualified and very familiar with the subjects they were asked to take care of, found they did not have time to undertake it at all. The work was taken up, however, others were appointed in their places, and in thirteen instances reports have been prepared.

Now, it must be borne in mind that these reports are only provisional, and in many cases are imperfect and incomplete. It has been our idea that each author should include only the methods with which he was perfectly familiar, and which he could personally recommend—not always satisfactory, but in all cases the most satisfactory to his knowledge—and usually inherent errors in the methods are stated and the degree of excellence is given. In several cases a great deal of time has been devoted to the study of methods by the various referees, often covering several months, so that several of the papers presented represent a fairly thorough study of the subject. In other cases, owing to lack of time, the referees have attempted to cover but part of their subjects, and it is intended, of course, that other portions shall be added later. In several instances—four, I believe—no report has yet been presented, because it was impossible for those in charge of the work to consider it sufficiently to offer reports. One report which has not been received yet will be prepared—that on infants' and invalids' foods—and published with the others. In all, 106 printed pages have been prepared. As each set of methods was completed it was distributed to a mailing list of 106, including each experiment station and agricultural college in the country, and, as far as we were acquainted with them, to every board of health and dairy and food-commission laboratory. Ordinarily it has been our custom to send but one copy to each laboratory, with the request that the recipient bring it to the attention of his associates. The result has not been all that we hoped. The criticisms have not been as extensive as we desired, and yet a number of minor changes have resulted from them, and these have been very helpful.

The committee met day before yesterday and yesterday to consider the full subject in as great detail as possible. I think there were only two or three methods—and those minor and unimportant—that were stricken out. In one instance this was due to the fact that since the methods were offered in the first place new work has been done and published by others and the methods there suggested taken up by the referee in charge, confirmed, found more satisfactory than his own, and substituted. In one other case it was thought by practically all of those who met and considered the matter that one of the methods given could be substituted by a somewhat similar one to advantage, and, since there was no objection to that, it was done. I think there was no method of those suggested cut out altogether.

We have put together here temporarily the methods that were distributed for criticism. The work was done as quickly as possible, with very little revision, the idea being merely to get them in shape for criticism. The proof was not read carefully, and there was a large number of typographical errors. Of course, all that will be corrected in the permanent report. It is not the idea that these shall be preserved, but merely criticised, and that the amended work shall be published in the near future.

I would call attention especially to one feature, which we regard as important, in which these methods differ from the regular methods of the association, and that is that in nearly all cases we have given the original reference for each method, with the name of the originator of the method. In some cases it was difficult to find the author of the method, but this was done as far as possible, and the committee thinks it would be an excellent plan to follow throughout the publications of the association.

One word regarding the practice of distributing these reports. It seems to me that it might be adopted to advantage by all of the referees of the association. All referees' reports could probably be completed a month earlier than they are. As a matter of fact very often they are not handed to the secretary until a month or two after the time of meeting. Now, if they were printed and distributed before the meeting of the association they could be discussed intelligently.

With regard to the correspondence that has come in I can say that we have been very much gratified by getting in touch with a great many chemists who were interested in food work whom we did not know at all, and we seem to have been brought into closer cooperation with them, and I believe it has added to this branch of the work of the association.

In closing I wish to express my gratitude to all of my associates for the hearty and cordial cooperation which I have received throughout, and while it is very difficult to particularize or specify, perhaps it is only due to the local associates, Messrs. Munson and Tolman, to recognize also their cooperation in the work in getting the reports ready for the printer, in addition to their own contributions, which were important, We have two criticisms or recommendations to make. In the first place, that the referee be known as the "referee on food adulteration," instead of on "liquor and food adulteration," as he is now called. You will recognize the fact that the liquor work is now a comparatively unimportant portion of the work of the referee, and there is no reason for specifying it in his title. Second, that the methods outlined by the referee and associates—as amended in our conference yesterday—be published separately as provisional methods of the association, under the authors' names.

The suggestions received in correspondence, when they are not adopted by the referees and the methods changed accordingly, will be printed in the publication referred to above as an appendix. Very often these suggestions are of considerable importance; new methods may be suggested which are considered of value, and yet if the referee has not tried them himself they can not well be included in his report. In some cases entirely new methods have been outlined. These may be of value, but we do not feel that we should assume the responsibility of them.

The PRESIDENT. Are there any special papers to be presented on the subject? We can easily imagine that this outline contains about everything that can be given at present. Discussion is in order on the subject. Is there anything more? If not, we will have the report on nitrogen.

Mr. Ferris then read the report on nitrogen, by Mr. Perkins.

REPORT ON NITROGEN.

By W. R. PERKINS, *Referee.*

The work of the referee during the past year has embodied only a repetition of the trials of the neutral permanganate (Street) and alkaline permanganate (Jones) methods for determining the availability of organic nitrogen.

Samples of fish, tankage, garbage fertilizer, dried blood, cotton-seed meal, and a mixed fertilizer the nitrogenous portion of which was furnished from cotton-seed meal, blood, and garbage constituted the samples distributed for the work.

In compliance with the request from different laboratories sixteen sets of these samples were sent out. Reports were received from eight laboratories embracing the work of twelve analysts.

The following letter, describing the samples and giving directions for the work, was sent out with the sample:

AGRICULTURAL COLLEGE, MISS., *June 27, 1901.*

DIRECTIONS FOR WORK ON NITROGEN FOR THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1901.

Six samples have been prepared for the work on nitrogen, as follows: No. 1, fish; No. 2, tankage; No. 3, garbage fertilizer; No. 4, dried blood; No. 5, cotton-seed meal; No. 6 is a mixture containing 100 parts of dried blood, 200 parts of cotton-seed meal, 600 parts of garbage, and 900 parts of acid phosphate.

TOTAL NITROGEN.

Determine total nitrogen by Kjeldahl method.

AVAILABLE NITROGEN.

Determine by the following methods:

(1) *Neutral permanganate method* (*Street*).—Into a 400-cc Griffin beaker, low form, weigh an amount of sample containing approximately 0.075 gram of nitrogen. Digest this with 125 cc of permanganate of potash solution (16 grams of potassium permanganate to 1,000 cc of water) in a steam or hot-water bath for thirty minutes. Have

the beaker let down well into the steam or boiling water and keep closed with a cover glass, stirring once or twice with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water, and filter through a heavy 15-cm folded filter. Wash with cold water till total filtrate amounts to 500 cc. Dry and determine nitrogen in residue by Kjeldahl method. Wash sample No. 6 before digestion, as in determining water soluble phosphoric acid.

(2) *Alkaline permanganate method* (*Jones*).—Weigh out an amount of sample containing approximately 0.045 gram of nitrogen, and transfer to a 600-cc distillation flask. After connecting with condenser to which the receiver containing standard acid has been attached, digest with 100 cc of alkaline permanganate solution (16 grams of potassium permanganate and 150 grams of sodium hydrate dissolved in 1,000 cc of water) for 30 minutes below the boiling point. Then boil slowly until the distillation is completed.

W. R. PERKINS, *Referee.*
FRED W. MORSE, *Associate Referee.*

The results of the work by the different analysts appear in the tables below:

TABLE I.—*Total nitrogen.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. B. Robb, College Park, Md	8.42	7.53	2.93	13.62	6.39	2.40
C. H. Jones, Burlington, Vt	8.34	7.40	2.91	13.56	6.30	2.38
C. D. Holley, Orono, Me	8.35	7.51	2.96	13.71	6.40	2.48
O. W. Knight, Orono, Me	8.38	7.48	3.02	13.69	6.42	2.46
T. M. Price, College Park, Md	8.58	7.49	3.06	13.74	....	2.45
J. W. Ames, Wooster, Ohio	8.30	7.56	2.98	13.81	6.41	2.57
W. R. Perkins, Agricultural College, Miss.	8.34	7.41	3.01	13.59	6.35	2.46
E. B. Ferris, Agricultural College, Miss	8.30	7.47	3.02	13.56	6.37	2.44
W. H. Scherffius, Lexington, Ky	[a] 8.23	[a] 7.38	[a] 2.93	[a] 13.56	[a] 6.36	[a] 2.40
Do	[b] 8.35	[b] 7.47	[b] 3.04	[b] 13.75	[b] 6.45	[b] 2.45
J. P. Street, New Brunswick, N. J	8.22	7.48	2.99	13.44	6.31	2.54
F. B. Carpenter, Richmond, Va	8.34	7.50	3.06	13.70	6.37	2.42
S. H. Shieb, Richmond, Va	8.36	7.49	3.08	13.69	6.43	2.35
Average	8.35	7.47	3.00	13.65	6.37	2.44

[a] Digested three hours. [b] Digested seven hours.

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. B. Robb, College Park, Md	0.66	0.38	1.43	0.29	0.34	0.60
	.70	.41	1.42	.25	.31	.70
	.60	.38	1.46	.39	.21	.70
C. H. Jones, Burlington, Vt	.85	.83	1.49	1.78	.91	1.05
C. D. Holley, Orono, Me	.89	.54	1.62	.96	1.37	1.30
	.89	.56	1.62	.96	1.37	1.30
	.89	....	....	1.00	....	....
O. W. Knight, Orono, Me	1.09	.69	1.71	1.06	1.39	1.46
	1.23	.70	1.71	1.02	1.43	1.38
	1.23	.70	....	.95	.37	1.35
	....	....	....	.95	....	1.40
T. M. Price, College Park, Md	.71	.54	1.54	.78	....	.81
J. W. Ames, Wooster, Ohio	.19	.46	1.61	.25	.33	1.01
W. R. Perkins, Agricultural College, Miss.	.80	.63	1.61	.96	.79	.71
	.64	.41	1.68	.81	.82	.73
	.67	.74	1.52	.64	.85	.70
	.84	....	1.54	.74	.86	.65

TABLE II.—*Results on digestion of organic nitrogen with neutral permanganate of potash*—Continued.

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
W. R. Perkins, Agricultural College, Miss.	....	....	1.61	....	0.65	0.41
	....	....	1.62	....	....	.40
	....	....	1.58	....	....	.56
	....	....	1.54	....	....	.51
E. B. Ferris, Agricultural College, Miss	0.67	0.54	1.56	0.59	.52	.59
	.59	.57	1.50	.47	.71	.56
	....	.53	....	....	....	.47
W. H. Scherffius, Lexington, Ky	.60	.32	1.51	.36	.46	.80
	.91	.35	....	.48	.41	.60
J. P. Street, New Brunswick, N. J	.82	.55	1.69	1.03	1.07	.65
	.84	.60	1.72	1.07	1.12	.67
S. H. Shieb, Richmond, Va	.64	.52	1.83	.48	.79	.84
	.67	.52	1.87	.66	.77	.82

TABLE III.—*Availability of organic nitrogen by the neutral permanganate method.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. B. Robb, College Park, Md	92.06	94.95	51.19	97.87	94.67	73.33
	91.68	94.55	50.85	98.16	95.30	70.83
	92.87	94.95	50.17	97.13	96.24	....
O. H. Jones, Burlington, Vt	89.75	88.76	48.59	86.86	85.06	55.97
C. D. Holley, Orono, Me	89.34	92.81	45.27	93.00	78.60	[a] 47.58
	89.34	92.53	45.27	93.00	78.60	[a] 47.58
	89.34	....	....	92.71	....	....
O. W. Knight, Orono, Me	86.99	90.64	43.37	92.25	78.34	[a] 40.65
	85.32	90.77	43.37	92.55	77.72	[a] 43.90
	85.32	90.77	....	93.06	78.66	[a] 45.12
	....	....	....	93.06	....	[a] 43.09
T. M. Price, College Park, Md	91.72	92.79	49.67	94.32	....	66.93
W. R. Perkins, Agricultural College, Miss.	90.40	91.50	45.51	92.93	87.55	[b] 71.13
	92.32	91.46	44.18	91.03	87.08	[b] 70.32
	91.86	90.01	49.50	95.29	86.61	[b] 71.54
	89.92	....	48.83	94.55	86.45	75.57
	....	....	46.51	....	89.76	[c] 83.33
	....	....	46.17	....	....	[c] 83.73
	....	....	47.50	....	....	[c] 85.36
	....	....	48.83	....	....	[c] 79.26
J. W. Ames, Wooster, Ohio	97.68	93.96	45.98	98.19	94.86	60.78
E. B. Ferris, Agricultural College, Miss	91.92	92.77	48.34	95.64	91.83	75.81
	92.89	92.36	50.33	89.16	88.85	77.04
	....	92.90	....	....	....	....
W. H. Scherffius, Lexington, Ky	92.81	95.71	50.32	97.38	92.86	67.34
	89.10	95.31	....	96.50	93.64	75.51
J. P. Street, New Brunswick, N. J	90.00	92.60	43.20	92.30	83.20	74.40
	89.70	92.00	42.10	92.00	82.20	73.60
S. H. Sheib, Richmond, Va	92.34	93.05	40.58	96.49	87.71	64.25
	91.86	93.05	39.28	95.18	88.02	65.10
Average	90.69	92.74	46.50	94.14	91.09	69.85

[a] Omitted from the average.
[b] Filter paper digested with sample.
[c] Sample washed off of paper with permanganate solution; omitted from the average.

TABLE IV.—*Results on digestion of organic nitrogen with alkaline permanganate of potash.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. B. Robb	5.72	5.58	....	6.42	3.64	1.42
	5.70	5.64	....	6.34	3.82	1.39
	5.78	5.60	....	....	3.80	1.43
C. H. Jones, Burlington, Vt	5.10	4.30	0.51	8.99	2.72	.71
C. D. Holley, Orono, Me	5.59	4.70	.66	8.56	2.58	.77
	5.59	4.70	.65	8.56	2.58	.72
	5.59	4.73	....	....	....	.72
O. W. Knight, Orono, Me	5.51	4.68	.60	8.33	2.57	.77
	5.43	4.58	.59	8.39	2.57	.75
	5.47	4.58	....	8.39	....	.71
T. M. Price, College Park, Md	5.54	4.14	.68	6.49	....	.72
W. R. Perkins, Agricultural College, Miss	5.65	4.92	.67	9.30	2.98	.71
	5.67	4.90	.73	9.54	3.16	.86
	5.63	4.87	.72	9.17	2.98	.78
	5.50	....	.69	9.35	2.96	.81
	5.78	....	....	9.04	2.64	.86
	....	....	....	....	....	.88
E. B. Ferris, Agricultural College, Miss.	5.77	5.25	.74	9.04	3.22	.79
W. H. Scherffius, Lexington, Ky	2.91	3.19	.77	7.28	4.34	.60
	4.76	4.04	.47	8.40	2.91	.60
	3.29	2.94	....	6.87	2.21	.43

TABLE V.—*Availability of organic nitrogen by the alkaline permanganate method.*

Analyst.	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
J. B. Robb, College Park, Md	68.01	74.10	....	47.13	56.96	[a] 59.16
	67.77	74.90	....	46.54	59.46	[a] 49.58
	68.40	74.36	....	....	57.78	[a] 57.91
C. H. Jones, Burlington, Vt	61.15	58.17	17.73	66.30	43.17	29.79
C. D. Holley, Orono, Me	66.94	62.58	22.29	62.44	40.31	31.05
	66.94	62.58	21.96	62.44	40.31	29.03
	66.94	62.97	....	....	....	29.03
O. W. Knight, Orono, Me	65.75	62.57	19.86	60.85	40.03	31.30
	64.80	62.58	19.54	61.29	40.03	30.49
	62.27	61.23	....	61.29	....	28.86
T. M. Price, College Park, Md	64.56	55.27	22.22	[a] 47.23	....	29.38
W. R. Perkins, Agricultural College, Miss	67.74	66.39	21.30	68.43	47.20	28.40
	67.98	66.12	24.30	[a] 72.47	50.00	30.50
	67.50	65.72	22.92	67.47	47.20	32.92
	65.95	....	23.92	68.80	46.61	34.95
	69.30	....	....	66.52	41.57	35.77
	....	....	....	....	....	32.52
E. B. Ferris, Agricultural College, Miss.	69.51	70.28	24.50	66.66	50.54	32.37
W. H. Scherffius, Lexington, Ky	[a] 34.80	[a] 42.59	[a] 25.00	[a] 53.10	[a] 67.49	[a] 24.49
	[a] 56.93	[a] 53.93	[a] 15.26	61.34	45.25	[a] 24.49
	[a] 39.35	[a] 39.25	....	[a] 50.16	[a] 34.37	[a] 17.55
Average	66.20	63.74	21.86	64.30	44.35	31.09

[a] Omitted from the average.

TABLE VI.—*Computed and determined availability of nitrogen in sample No. 6.*

	Total N.	Neutral permanganate method.		Alkaline permanganate method.	
		Undissolved N.	Availability of N.	N. removed.	Availability of N.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Computed	2.46	0.82	66.66	1.02	41.46
Determined	2.44	.74	69.85	.76	31.09

COMMENTS BY ANALYSTS.

J. M. Bartlett.—From the study of results I think that the neutral permanganate method promises much better than the alkaline permanganate; although, perhaps, comparative results with the alkaline show as great differences as the neutral, still the percentages of available nitrogen are not nearly up to what soil tests show.

J. B. Robb.—In the alkaline permanganate method I added some pumice stone before distilling to prevent the contents of the flask from bumping.

John Phillips Street.—I followed your directions in every particular, except that I was contented to stop with 400 cc of washings. Past experiments which I have made show that there is a very small amount of nitrogen in the total filtrate, by far the greater part being driven off by the permanganate during the digestion. The prolonged washing suggested, therefore, seems to be unnecessary. Four or five thorough washings should suffice.

F. B. Carpenter.—Mr. Sheib reports that he was unable to get any satisfactory results by the alkaline permanganate method. He experienced great difficulty in distilling the nitrogen after digestion on account of violent bumping, and in some instances excessive foaming. The neutral permanganate method seemed to give very much more satisfactory results in case of the class of materials which were included in your samples. I do not believe, however, it is of very much value as a standard for comparison with the actual availability of the compounds in the soil. I have seen some results by this method on organic ammoniates which had been acidulated with sulphuric acid which were lower than the material showed before the treatment.

A. M. Peter.—In regard to the total nitrogen you will notice that there is a perceptible gain by digesting a longer time. It would seem that six hours is the time needed for these samples. While the average fertilizer may give up its nitrogen in about three hours' digestion, I do not think that substances high in nitrogen will always do so, and I think our official directions ought to be so modified as to call attention to this point, which is quite likely to be overlooked. It will not always do to stop the digestion when the solution has become colorless, or nearly so, or a light straw color. I believe this point has been worked on over in Germany, and the evidence obtained is somewhat conflicting, though much of it tended to show that long digestion was necessary where extreme accuracy is required, and that the use of P_2O_5 in the sulphuric acid hastens the results. An experiment I made some time ago upon tobacco stems may be of some interest in this connection, though involving the method as modified to include nitrates. I used 1.4 grams of stems, 0.5 gram of salicylic acid, 30 cubic centimeters sulphuric acid, 2.5 grams of sodium thiosulphate, and 0.5 gram metallic mercury.

The following percentages of nitrogen were obtained:

	Per cent.
1. Digestion as usually done here, not timed, average of 2	2.60
2. Digestion stopped as soon as colorless	2.43
3. Digestion continued much longer, not timed	2.71
4. Digestion stopped at end of 2 hours, mean of 2	2.74
5. Digestion stopped at end of 3½ hours, mean of 2	2.80
6. Digestion stopped at end of 5 hours, mean of 2	2.82

In this instance it seems the digestion had to be continued considerably after becoming colorless, and required about three and one-half hours.

DISCUSSION OF RESULTS.

No comments on the results in Table I are necessary, further than calling attention to the differences existing between the highest and the lowest determinations on samples 1, 4, and 6. The results sent by Dr. Peter, together with his comments, indicate a probable cause of such variation. My own observations on such material as blood, fish, and cotton-seed meal have led me to the same conclusion, and I suggest the advisability of placing such a precaution in the method, or else that the matter should receive the attention of the association, through its referee, on this subject. By reference to Table II we observe that with samples Nos. 1, 2, and 3 the figures are comparatively uniform, with the exception of four results. In samples with as high a percentage of nitrogen as Nos. 1 and 2 contain, such variations do not count for so much on the per cent of nitrogen available, and on the whole, I think, are quite satisfactory. With samples Nos. 4, 5, and 6 the differences are more marked, but also assume very satisfactory proportions after rejecting a few of the higher ones. With regard to this method we will note two especial conditions in the manipulation, one or both of which may account for the varying results in this table.

1. After digesting and filtering the sample should be washed to a uniform extent.
2. All fertilizers containing acidulated material should be washed till free of acid before digestion. It is undoubtedly true that much of the nitrogen by this method remains in the filtrate and is not converted into ammonia by the permanganate. This being the case, it is evident that insufficient washing will leave some of the filtrate in the paper or sample and consequently increase the amount of undissolved nitrogen. As evidence on this point, mention was made in the report of last season of the fact that the soluble phosphoric acid of acid phosphate would precipitate nitrogenous compounds from the filtrates. Further work has shown that acids, both organic and mineral, will act in the same manner, throwing down a copious precipitate of these protein compounds. As bearing further on this point, a full set of the official samples were digested as usual, and instead of adding 100 cc of water before filtering, 100 cc of 1 per cent hydrochloric acid was added, and the filtration and washing continued as usual. The nitrogen in the results was as follows:

TABLE VII.—*Digestion of organic nitrogen with neutral permanganate of potash, and additions of hydrochloric acid.*

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	No. 6.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Nitrogen in residue	3.00	2.80	1.90	4.15	4.26	1.67
Nitrogen removed	5.31	4.51	1.11	9.44	2.09	.79
Total nitrogen removed	63.95	60.37	37.00	69.15	32.18	32.37

The results in the first trial are very much higher than those obtained in the regular way. In fact, the portions of nitrogen removed by the process bear a very striking resemblance to the so-called available nitrogen by the alkaline permanganate method as shown in Tables IV and V. Mention is made of this in order to emphasize the importance of uniformity in washing the residue, and also to call attention to the results obtained on acidulated goods if the free acid is not removed before the digestion of the sample with permanganate. The results by the alkaline permanganate method are very unsatisfactory when considered as a whole, though an examination of the results of the different analysts, with one exception, indicates that entirely satisfactory results might be obtained by the method. From my own experience with the method I felt very much encouraged as to its reliability in giving concordant results, but a study of Tables IV and V emphasizes the fact that this method is sensitive (even more so than the neutral permanganate method) to changes in conditions governing its execution. The permanganate should be pure, the quantity exact, and the expression "below the boiling point" in the directions for the preliminary digestion should be changed to words more definite in meaning. In my own work I adopted as the temperature for the digestion a point just below that at which the contents of the flask would boil. The heat was so regulated as to bring them to this point in about ten minutes, after which they were kept not more than one or two degrees below boiling till the expiration of the thirty minutes, when the lights were turned up and the distillation completed in the time ordinarily required for a nitrogen distillation. No serious bumping or foaming of the contents of flasks was experienced in any of the work.

From the ease and rapidity with which this method can be executed, together with the individual results reported by the different analysts, I think it worthy of further trial. The most unsatisfactory feature connected with it seems to be its failure to place cotton-seed meal in the relative position indicated for it by vegetation experiments.

CONCLUSIONS.

While the results by the neutral permanganate method do not agree perfectly, they are sufficiently uniform to render the method of value in distinguishing between the different grades of nitrogenous material as determined by vegetation tests. This method places the availability of organic nitrogen at a much higher figure than is obtained in soil tests, but from a comparison of the results on all the materials that have been tested by it during the past four or five years, with the results obtained in pot experiments, there is a very satisfactory agreement in the relative position of their nitrogen availability.

The availability of nitrogen in materials of the first class is well up into the eighties and nineties, while garbage, leather, and such materials are well under 50, making quite a gap between them and rendering the detection of adulteration easier. Since about 40 per cent of the nitrogen in sample No. 6 is derived from garbage, it is apparent that a fertilizer, the determined availability of the nitrogen of which falls below 70 per cent, would be considered as of doubtful quality.

I hesitate to recommend the adoption of this method by the association in its present form, but, feeling that reliable results may be obtained by it when carefully executed, will give it to the association as follows:

Into a 400 cc Griffin beaker, low form, weigh an amount of sample containing, approximately, 0.075 grams of nitrogen (samples containing material that has been treated with acid should be washed on a 9 cm S. S. No. 595 filter to 200 cc and transferred, filter and all, to beaker); digest this with 125 cc of permanganate of potash solution (16 grams of pure $KMnO_4$ to 1,000 cc of water) in a steam or hot-water bath for thirty minutes. Have the beaker let down well into the steam or hot water and keep closed with a cover glass, stirring twice at intervals of ten minutes with a glass rod. At the expiration of the time remove from bath, add 100 cc of cold water and filter through a heavy 15 cm folded filter. Wash with cold water, small quantities at a time, till total filtrate amounts to 400 cc. Dry and determine nitrogen in residue by Kjeldahl method.

RECOMMENDATIONS.

It is recommended—

(1) That the neutral permanganate method be adopted as a provisional method.

(2) That the alkaline permanganate method be further tested by the succeeding referee.

(3) That a caution be inserted in the method for total nitrogen concerning the proper digestion bath of sulphuric acid with certain classes of material, or that the question be investigated by the referee.

The PRESIDENT. Are there any special papers relating to the determination of nitrogen, or any discussion of the paper presented?

Mr. Wiley is very desirous that some important work which has been done on cider by Mr. W. B. Alwood should be brought before the association, as several members of this body are interested in that line of work, and Mr. Alwood has kindly consented to make a brief statement in regard to it. I take this time to give him the opportunity of doing so.

ADDRESS OF MR. W. B. ALWOOD.

MR. PRESIDENT AND GENTLEMEN OF THE CHEMISTS ASSOCIATION: I am somewhat at a loss on my first appearance before a body of chemists, and especially such a dignified body of official chemists.

After hearing your president's address yesterday there is a question in my mind as to the specific gravity of a horticulturist or worker in economic botany. But I am happy to say that I have always been able to work in the most pleasant official relations with my brother chemists, and whenever I want to know anything about the constituent parts of any substance, I go to the chemist. In fact the whole data for the control of life problems I secure from the chemical laboratory.

Hence, after hearing your president's encouraging remarks on the importance to chemists of the work in the great field of life problems, it leads me to believe you will be interested in a work which I took in hand some years ago. This work was in a way forced upon me by its economic importance to fruit growers. And in the fewest words possible, it comprises methods of utilizing our unmerchantable fruit.

The fruit crop of the United States in the aggregate is enormous. Crop statistics generally give us in the case of apples only the merchantable crop, and in many cases only that which enters into foreign commerce. I have no adequate idea of the quantity of unmerchantable fruit produced in a crop year in the United States, but from statistics gathered in Virginia it is in my opinion safe to say that in a good crop year we have about 400,000 bushels of apples alone which fall below the best market grade.

I feel certain that anyone who has observed conditions in the great orcharding sections of our country will approve the statement that we simply do not know how much fruit of inferior grades goes to waste. And this is not true only of apples. How often does it happen that grapes are scarcely worth the expense of picking and marketing. I was told only this week of an instance where 400 tons of grapes were heaped up at a single New York cellar waiting to be used up in wine making. This implies that even first-class merchantable fruit may become a waste product because it can not be marketed.

I have said waste product not unadvisedly, because though we manufacture many products from our unmerchantable and unmarketable fruits, we ordinarily waste the bulk of this material by reason of bad methods, or by making a product which is worse than waste.

To illustrate, the 400 tons of grapes mentioned above, were said to be piled up in mass, with the juice oozing out and dripping from them. This state of affairs indicates not only a loss of the must which thus runs away, but offers the best possible opportunity for all sorts of mal-germs to enter and begin their destructive work on the sugars of the fruit, and to contaminate the mass with flavors which can not afterwards be eliminated.

We heap up great quantities of inferior apples on the ground and then leave them to sweat and rot, thus destroying their intrinsic worth in sugar content, and allowing them to become seed beds for mal-organisms and to acquire in addition earthy flavors which can not be gotten rid of either in cider or other products made therefrom.

We also work inferior apples into sun-dried fruit in the South in great quantities. Two hundred tons of this product is often shipped in one year from our little depot at Christiansburg. This product returns as a gross price, 5 to 7 cents per bushel of apples to those who make it. Is this not an enormous waste of labor, let alone material? And it serves to put upon the market a product which injures the sale of better goods.

Now, the question arises, What shall we do with this refuse or unmerchantable fruit? Canning, marmalade, etc., will consume an always increasing portion of it, but is there not room for a study of cider and vinegar making? This question has been thrust upon me in Virginia, and for the past eight years I have been working upon it with a view to reducing the methods and technique to a scientific statement.

First of all, I needed the chemical data on the composition of our fruits, and the changes which they undergo either in bad storage or improper fermentation processes. This data in American literature is very meager, though receiving increasing attention at your hands. Critical chemical data is absolutely necessary before I can, as a student of the fermentation processes, reach any conclusions of value.

Then also the question of the specific organisms which induce fermentation presents a study of the most vital importance, and it is on this phase of the subject that I have concentrated my part of the work. The American literature on this side of these questions is even more meager than on the chemical side. After seven years of work and experiment, I concluded that there was no better way to master the details of this subject than to go abroad and work it up in the foreign laboratories, where such leaders as Wortmann, Hansen, Linder, Fernbach, Kayser, and others had given much study to these subjects. This I was able to do through the generous assistance of Dr. Wiley, Chief of the Bureau of Chemistry, and through his continued assistance we have now established at Blacksburg an œnological laboratory for the continuance of this work.

In this laboratory we have already brought together a representative collection of the alcoholic ferments, viz, the true yeasts of the group Saccharomyces which are used in Europe for the fermentation of some of the most noted wines and ciders, and also a collection of mal-ferments, and some of the acetic acid ferments. These latter fall correctly under the Bacteriaceæ, being known to botanists as *Bacterium aceti*. The use of the term Mycoderma to designate vinegar ferments is incorrect, though it occurs in the American literature up to the present date.

In addition to yeast races purchased abroad, I have also isolated a number of such races from material collected in Europe, and am now engaged on American material.

This seems to me a most promising field. While I do not for a moment believe the wide claims made in some quarters as to the efficacy of special yeast races in producing with certainty the desired characteristics in a wine or cider, I know from my own work that these races when used in pure cultures in a proper manner will produce markedly different results, some desirable and others undesirable. Now, what we need to know is how to determine these facts with certainty, how to isolate and cultivate the desirable races in a commercial manner, and to devise a reliable technique for their practical use.

That color of the liquor, alcoholic strength, boquet, and flavor are affected by the organisms which produce the fermentation, is fully settled; but the "how" to surely bring about in a practical manner these desirable conditions and qualities is not settled by a long way.

We are however arriving at some knowledge of how the multiplicity of organisms, always present in any normal must, behave toward the same, and the conditions which favor or hinder their vital activities, and it is on these lines that the great field of life chemistry here opens up before the artisan who deals with these forces to produce a practical result.

I can not go into details in the few moments allotted to me, but I wish to repeat that we know enough at present to say that the key to good practical results appears to lie quite largely in the direction of pure cultures of strong yeast races which are capable of producing a sound fermentation, and taking possession of the must to the practical exclusion of mal-organisms. There is, of course, a great amount of detail as to cleanliness and temperature conditions and technique of handling the liquor. These will be treated in my reports to the Bureau of Chemistry.

The collections of yeast races and other organisms I have mentioned will, I believe, be put in shape for general distribution to specialists who may desire them. This matter rests with Dr. Wiley, and he should be consulted about it.

I am authorized to say that we need and earnestly request cooperation on the part of all the members of this association. The first desideratum is chemical data on all our fruit products, either fresh fruits, fruit must, wines, ciders, vinegars, etc. This part of the work in America presents a herculean task, and while Dr. Wiley's bureau is at work upon it, and Professor Davidson of our station is working on the chemistry of fruits and by-products of fruit, there is room for all, and we wish to compile and publish in this study as complete results as are attainable from all American sources.

Pardon a worker on the economic side for advising the chemists, but in your fruit analyses I wish to say that the next important ingredient to sugar and acid content is the tannin. This constituent of a fruit must is of the utmost importance in clarifying and assisting in other details of sound fermentation, hence it should always be determined.

Now, in conclusion, let me say this is not a small subject. It involves a study of the entire question of the preparation of alcohol in all its aspects, whether in beverages or liquors, for use in arts, manufactures, or science, and incidentally the preparation of all truly normal vinegar products.

I thank you for your indulgence.

The PRESIDENT. I am sure we have all been glad to hear this report, and some, who have done work on the subject of cider vinegar, are more than ordinarily interested in these statements and will look forward with a great deal of hope and expectation to the future development of this work.

Mr. ALWOOD. I did not touch vinegar. There also we are working out the races of the germ *Mycoderma aceti*, which is the true vinegar germ.

Mr. EWELL. Mr. President, before you take up the regular program I have a matter I would like to bring before the association. It is a suggestion for an amendment to the constitution. I bring it up inasmuch as a committee has been appointed for that purpose, and I would like to have you refer it to that committee. It refers to the question of membership in section 2. The part which I propose to strike out is as follows:

> Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution.

This part of the section has never been adhered to, and as new members make inquiry concerning this point it rather complicates matters. I suggest the following in place of the words quoted above:

> All persons eligible to membership shall become members ex officiis and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials.

Referred to special committee on amendments.

The PRESIDENT. Next in order will be the report on insecticides.

Mr. HAYWOOD. Mr. President, the work on insecticides was divided into two parts, the arsenicals being given to the associate referee and the remainder of the subject to the referee. Professor Voorhees was not able to come to the meeting, but has sent his report. Mr. Street, however, was able to come down, and as he has been working with Professor Voorhees on this matter, I shall be very much obliged to him if he will read Professor Voorhees's report, to be followed by the report of the associate referee.

Mr. STREET. I have only slightly more familiarity with this work than Mr. Haywood, and have not even looked over the report, but I will read it.

INSECTICIDES AND FUNGICIDES.

By L. A. VOORHEES, *Referee.*

For reasons which were given at the time, the reports on insecticides and fungicides at the last two meetings of the Association of Official Agricultural Chemists embraced merely a consideration of proposed methods for various lines of work. This is the first year, therefore, in which any analytical work of a cooperative nature has been undertaken by the association. In order that the relative importance of the several lines of work, or, at least, the relative interest in the same might be discovered, requests, or invitations, to participate in all of the lines were sent to the members of the association in the shape of the following letter of inquiry:

DEAR SIR: Will you kindly inform me if you or any of your staff will cooperate in the work on insecticides this year; and if so, which of the following lines you are willing to take up:

Alkalinity in potash and soda lyes.
Arsenic in arsenicals.
Copper in copper carbonate.
Cyanogen in potassium cyanide.
Formaldehyde in formalin.
Nicotin in tobacco.

An early reply will be appreciated.

I remain, etc.

To this letter 48 replies were received, of which 26 were negative in their character and 22 affirmative. Many of the latter mentioned two or more lines in which they were interested, so that the number of individuals interested in each line was as follows:

Alkalies	6
Arsenic	14
Copper	9
Cyanogen	6
Formaldehyde	14
Nicotin	7

From this it will be noticed that equal interest was betrayed in arsenic and formaldehyde, and the interest in these two equaled the combined interest expressed in all the others.

Mr. J. K. Haywood, the associate referee, had very kindly consented to take complete charge of the work with the arsenic-bearing materials, an arrangement of which, in view of Mr. Haywood's experience in the study of the subject, the association can not fail to approve. To him the 14 names above alluded to were communicated, and from him you will hear the report of the work which has been done along that line. The writer himself took charge of the remaining lines.

As samples for the work in alkalies two commercial lyes were chosen, and with these were sent two samples of "whale-oil soap." The one was the product of James Good, of Philadelphia, and the other of Leggett and Brother, of New York City, both of whom kindly furnished an abundance of their product free of charge. To those who had expressed a wish to work with copper a sample of commercial copper carbonate was sent. Copper carbonate was sent also to each one to whom samples of paris green had been sent, with the request that copper determinations be made. As material to try the method proposed for cyanogen a sample of potassium cyanide was chosen. To those who desired to try the methods for formaldehyde a solution of formalin was sent, which was understood to run about 39 per cent of formaldehyde. To those who expressed a wish for the nicotin samples a tobacco extract and a tobacco powder were submitted. These samples were very generously supplied by Mr. R. G. Mewborne, chemist of the Kentucky Tobacco Product Company. With the samples were sent letters of instructions as to the methods and other particulars outlined in the proceedings of the last convention of the association.

Your referee regrets to report that the work which has been done along the lines coming under his eye is very meager. He has received but eight reports in all, of which two are on copper, two on cyanogen, and four on formaldehyde. These reports, while few in number, comprehend considerable work, and it has been deemed but fair to give them in full.

FORMALDEHYDE.

A. W. Ogden, of the Connecticut experiment station, writes as follows:

> Inclosed you will find some results of determinations made on the referee's samples of formalin, and also on another sample of weaker formaldehyde, which we had on hand. In Table I are given the results by the Blank and Finkenbeiner method, and in Table II by the Legler, or ammonia method, as carried out in the following manner:
>
> About $1\frac{1}{3}$ grams of formalin were weighed into a 3 or 4 ounce flask, containing an excess of standard ammonia, tightly stoppered, and shaken occasionally for sixteen hours, when the excess of ammonia was titrated with normal sulphuric acid, using one or two drops of a 0.5 per cent solution of corallin as indicator.
>
> Some determinations were made by heating the mixture of formalin and ammonia in boiling water for one hour, cooling and titrating the excess of ammonia with sulphuric acid. By the latter method, I obtained 37.72, 38.15 and 38.63 per cent on your sample, and 20.38 and 20.94 on ours.

I see no advantage in heating, providing the mixture of formalin and ammonia is allowed to stand from twelve to sixteen hours in the cold. Where it stood two hours or less, as in determinations 1 and 2 in Table II, the results were low.

It took but a few minutes to make a determination by the Blank and Finkenbeiner method, while the results agree on the average with those by the ammonia method.

TABLE I.—*Referee's sample of formalin, 1901.*

[Blank and Finkenbeiner method.]

No.	Elapsed time between addition of H_2O_2 and titration.	Formalin.	2N-NaOH.	2N-H_2SO_4	2N-NaOH combined as CHO_2Na.	Formaldehyde (CH_2O).
	Minutes.	*Grams.*	*cc.*	*cc.*	*cc.*	*Per cent.*
1	3	1.3910	25.00	16.60	8.40	36.23
2	5	1.3357	25.00	16.50	8.50	38.18
3	5	1.1590	25.00	17.60	7.40	38.31
4	5	1.0753	25.00	18.15	6.85	38.22
5	5	1.1716	25.00	17.55	7.45	38.15
6	5	2.8859	30.00	12.20	17.80	37.06
7	8	3.0394	30.00	10.90	19.10	37.70
8	10	2.9854	30.00	11.30	18.70	37.58
9	10	2.1775	25.00	11.30	13.70	37.75
10	15	2.9620	25.00	6.40	18.60	37.68

RESULTS ON ANOTHER SAMPLE OF FORMALDEHYDE.

1	5	1.1825	25.00	20.90	4.10	20.80
2	5	1.3057	25.00	20.50	4.50	20.68
3	10	3.5635	25.00	12.80	12.20	20.54
4	10	.2740	25.00	24.00	1.00	21.90
5	10	.5423	25.00	23.05	1.95	21.58

TABLE II.—*Referee's sample formalin, 1901.*

[Legler, or ammonia, method.]

No.	Elapsed time between addition of NH_4OH and titration.	Formalin taken.	Standard ammonia (1 cc=0.004857 grams NH_3).	N. sulphuric acid (1 cc=0.017 grams NH_3).	Ammonia (NH_3) combined as $(CH_2)_6N_4$.	Formaldehyde (CH_2O.)
	Hours.	*Grams.*	*cc.*	*cc.*	*Gram.*	*Per cent.*
1	½	1.3215	50.00	3.70	0.1799	36.03
2	2	1.3662	50.00	3.40	.1850	35.84
3	16	1.3280	50.00	3.10	.1901	37.89
4	16	1.3011	50.00	3.34	.1860	37.84
5	16	1.3189	50.00	3.20	.1884	37.81
6	16	1.3157	50.00	3.20	.1884	37.90
7	16	1.3204	50.00	3.20	.1884	37.77
8	16	1.3707	50.00	2.70	.1969	38.02
9	16	1.1900	50.00	4.20	.1714	38.13

RESULTS ON ANOTHER SAMPLE OF FORMALDEHYDE.

1	16	2.0775	50.00	4.35	0.1688	21.51
2	16	1.8970	50.00	5.18	.1547	21.58

The following results were obtained by Mr. R. W. Thatcher, of the Washington Experiment Station:

METHOD I.—*Oxidation by H_2O_2 (Blank and Finkenbeiner)*

Weight of formalin taken.	2 N-NaOH used.	Required for 3 grams.	CH_2O.
Grams.	*cc.*	*cc.*	*Per cent.*
3.3832	13.97	12.39	24.78
3.1838	13.60	12.76	25.52
3.1957	13.82	12.97	25.95
3.1407	13.80	13.14	26.28
3.1165	13.80	13.25	26.50
3.1663	14.36	13.57	27.14
2.9372	14.06	14.36	28.72
2.9288	14.07	14.41	28.82
Average...	....	....	26.72

METHOD II.—*Weighing as hexamethylamine (Connecticut Station).*

Weight of formalin taken.	Weight $(CH_3)_6N_4$.
Grams.	
10.2400 9.9870	Not determined, because compound was still losing weight after five weeks' drying in desiccator over H_2SO_4.

METHOD III.—*Ammonia equivalent (Connecticut Station).*

Weight of formalin taken.	$N_{10}NH_4OH$ used.	CH_2O equivalent.	CH_2O.
Gram.	*Gram.*	*Gram.*	*Per cent.*
0.1841	0.02755	0.07273	39.49
.2458	.03587	.09470	38.50
.2403	.03590	.09477	39.41
Average...	....	....	39.13

METHOD IV.—*Iodometric method.*[a]

Weight of formalin taken.	Iodine absorbed.	CH_2O equivalent.	CH_2O.
Gram.	*Gram.*	*Gram.*	*Per cent.*
0.1073	0.35682	0.04222	39.33
.0537	.17979	.02127	39.43
.0537	.17915	.02120	39.49
.1341	.44751	.05307	39.57
.1610	.53037	.06275	39.00
Average...	....	....	39.36

[a] Allen's Common Organic Analysis, Vol. I, p. 220.

Method I gave entirely too low results. It is possible that 50 cc of my hydrogen peroxid was not sufficient to complete the oxidation. The fact that the larger the amount of formalin taken the lower was the per cent of CH_2O found might indicate this. Lack of material prevented my testing this point further. My observation would tend to cause doubt as to the reliability of the method as outlined at present.

Method II is far too slow for a practical method. Four weeks' drying in a desiccator, after evaporation at 75°, nearly to dryness, was insufficient to obtain constant weight.

Method III is easily carried out and gives fairly concordant results.

Method IV seems to me to be the most satisfactory one that I have tried. It gives very concordant results and is very quickly and easily carried out. It would be especially valuable for work with dilute solutions of formaldehyde.

Mr. A. E. Leach, of the food and drug laboratory of the Massachusetts State Board of Health, reports results by himself and by his assistant, Mr. Lythgoe, as follows:

Estimation of formaldehyde, Massachusetts State Board of Health.

	Weight of formalin taken.	2N NaOH required for weight taken.	Per cent formaldehyde CH_2O.
	Grams.	*cc.*	*Per cent.*
Leach, 1	3.2745	20.5550	37.66
Leach, 2	3.2365	20.3495	37.72
Lythgoe, 1	3.3220	20.8999	37.75
Lythgoe, 2	3.2284	20.5224	38.12

Our experience with the ammonia method is such that we have very little faith in its accuracy, referring especially to the method of evaporation. We find it almost impossible to get a constant weight, since loss of weight will continue as long as the hexamethylamine residue remains on the water-bath. I regret that we have not had opportunity to try the method of titrating the excess of ammonia with standard acid.

The method we prefer in this laboratory for estimation of formaldehyde is the iodometric method of Romijn.[a] According to this method Mr. Lythgoe obtained a result of 38.27 per cent on your sample. A possible explanation of these low results may be the fact that the samples were kept in the laboratory more than two months before they were analyzed.

The following results were secured in the laboratory of the New Jersey Experiment Station, by the Blank and Finkenbeiner method. Mr. Street had a separate sample, which was bottled at the same time and in the same manner as those which were sent out to the other analysts, and a similar sample was retained for the use of your referee. The work was performed independently with different standardized solutions, which were afterwards compared with each other and found to agree. The samples also were exchanged.

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[a] Zeits. anal. Chem., 1897, **36**, 18-24. Abstract Analyst, **22**, 221.

Estimation of formaldehyde (New Jersey station).

DETERMINATIONS BY MR. STREET.

Sample.	Weight of formalin taken.	2N NaOH required for weight taken.	Formaldehyde, CH_2O.
	Grams.	*cc.*	*Per cent.*
1. Street's sample	3.4459	22.49	39.16
2. Street's sample	2.5452	16.78	39.56
3. Street's sample	2.9057	19.18	39.60
4. Street's sample	2.8629	18.69	39.18
5. Street's sample	2.6992	17.76	39.48
6. Referee's sample	2.6458	17.31	39.25

DETERMINATIONS BY REFEREE.

1. Referee's sample	2.3645	15.42	39.13
2. Referee's sample	2.9774	19.50	39.30
3. Referee's sample	2.7040	17.68	39.23
4. Street's sample	3.1954	20.82	39.09
5. Street's sample	2.8566	18.59	39.05

COMMENTS.

A comparison of the results which have been reported shows that neither analyst had great difficulty in securing results by any one method, which agreed among themselves; the results by different methods were divergent, however. The lowest results by the hydrogen peroxid method are reported by Mr. Thatcher, but his results by the ammonia method and the iodometric method agree well with each other, and also with those secured by the hydrogen peroxid method by your referee and by Mr. Street. On the other hand, the results of Mr. Leach by the iodometric and hydrogen peroxid methods, and those of Mr. Ogden by the ammonia and hydrogen peroxid methods, are concordant, but all are about 1 per cent below the high results of Mr. Thatcher and those from New Jersey. If it is permissible here to use the axiom that things which are equal to the same thing are equal to each other, it would seem that no one of the methods is definitely proved to be worthless. The hydrogen peroxid of Mr. Thatcher may have been too weak, as he has suggested, and in the case of Mr. Leach and Mr. Ogden there may have been a depreciation in the sample. The method of weighing the portions taken for the determinations is also a matter of importance. The solution when exposed to the air gives off a strong odor of formaldehyde. That exposure occasions an undetermined but appreciable loss. In the work of your referee the portions for analysis were taken by weighing the formalin in a stoppered Schuster dropping bottle. Approximately 60 to 75 drops were then dropped directly into the standard sodium hydroxid, and the bottle was restoppered and weighed to show, by loss, the amount taken. Mr. Street, before this method was chosen, obtained results as low as 29.38 and no higher than 35.05 per cent. The strength and relation of the peroxid of hydrogen also has a bearing, for it was the observed fact with each of the analysts that the results varied inversely with the amount of formaldehyde to be oxidized. The time allowed to elapse before titration also seems to have had a bearing on the case.

The referee would like to add that the peroxid of hydrogen, which was used by Mr. Street and himself, was entirely free from acid, which is not the case with all samples of this product. Presence of acid would cause an apparent increase in the amount of formaldehyde present.

QUALITATIVE TESTS FOR FORMALDEHYDE.

With the samples of formalin, requests were sent to make tests of certain methods for its detection, qualitatively. Your reporter had no intention of trespassing on the field of the referee on food adulteration, or of his associate, who had the matter of preservatives in hand, and his apology is here tendered. The few results secured are presented, however, in order to deal fairly with the analysts who so kindly carried out the tests.

Mr. A. W. Ogden, of the Connecticut station, reports as follows:

A solution containing 1 part formaldehyde to 1,000 parts water was made by diluting 1.3 grams of the referee's sample of formalin to 500 cc with distilled water. To 10 cc of this solution were added 990 cc of milk, making a mixture containing 1 part formaldehyde to 100,000 parts milk. Good tests were obtained on this milk, using the following methods.

Hydrochloric acid test.[a]

Ten cubic centimeters of milk were shaken in a test tube with 5 cc of strong hydrochloric acid containing 1 drop of a ferric chlorid solution and gently heated, never to boiling, with constant agitation. The presence of formaldehyde was indicated by a purple color.

Sulphuric acid test.

To 5 cc of milk in a test tube an equal amount of strong sulphuric acid, containing a trace of ferric chlorid, was carefully added so the two did not mix; a violet or purple ring at the point of contact denoted the presence of formaldehyde. This test appeared more delicate when the milk was mixed with three or four times its volume of water, and three or four drops run carefully, to avoid charring, into a test tube containing 5 cc of the strong sulphuric acid mixture and 3 drops of ferric chlorid to 100 cc of acid.

Rimini test.[b]

One hundred cubic centimeters of milk, with one drop of a 50 per cent citric acid solution, and a piece of paraffin, were distilled until 20 cc had come over. To 15 cc of the distillate were added 1 cc of a solution of strong phenylhydrazine hydrochlorid (4:100), 4 drops of a freshly prepared solution of sodium nitro-prussid and 4 or 5 drops of a 50 per cent sodium hydroxid solution. Formaldehyde was indicated by a green coloration, which changed to red on standing. Fifteen cubic centimeters of distillate from a sample of milk, containing 1 part formaldehyde in 20,000 parts, gave with Rimini's test a beautiful blue coloration, which soon changed to green and finally to red.

Mr. R. W. Thatcher reported as follows:

I prepared solutions containing 1 to 10,000, 1 to 20,000, etc., up to 1 to 1,000,000 parts of formaldehyde and tested by the various reagents indicated below. In each case after applying the reagent to 10 cc of the solution, the mixture was allowed to stand fifteen minutes and at the end of that time I noted first, the limit to which the color change was sufficiently striking to afford what I would deem a reliable evidence of the presence of formaldehyde, and second, the limit to which any color change could be detected by comparing with a blank tube containing no formaldehyde. The following results were observed:

Qualitative determinations of formaldehyde with various reagents.

Author.	Reagent.	Limit of reliability.	Limit of sensibility.
Hehner	H_2SO_4 + trace if Fe Cl_3	No change in any case.	
Schiff	10 cc of fuchsin decolorized by SO_2	1 to 20,000	1 to 60,000
Do	Same after standing 1 hour	1 to 100,000	1 to 400,000
	Phenol pour into H_2SO_4	1 to 100,000	1 to 400,000
Lebbin	1 cc 5 per cent solution of resorcin 1 cc 5 per cent solution of NaOH	1 to 100,000	1 to 600,000
Vanini	5 drops 1 per cent solution of phloroglucin 5 cc 4 per cent solution of NaOH	1 to 20,0000	1 to 1,000,000

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[a] Conn. Agr. Expt. Sta. Rept., 1900, p. 127.

[b] Anal. di Farmacol, 1898, **97** (Abstract Zeitschr. Unters. Nahr. Genuss, **1**, 858.)

I would recommend that the Hehner test be taken from the list of official methods, as it is very unsatisfactory and unreliable with even moderately dilute solutions. The Schiff's reagent gives the most striking and most easily perceptible color change, but is not sensitive to very small quantities of formaldehyde, unless the mixture is allowed to stand for one hour at least. I found the phloroglucin test, as recommended by Vanini, to be the most satisfactory of any that was tried, but the pink color is not very pronounced and not so easily seen as some of the others.

Mr. A. E. Leach reported as follows:

We have had occasion many times to try the qualitative tests for formaldehyde. In our regular milk work we test all samples for preservatives during the months of June to September, inclusive, amounting to about 20 samples per day. The preliminary test that we have adopted as being by far the most satisfactory, is that of heating below boiling with hydrochloric acid and ferric chlorid.[a] This is much more delicate than the sulphuric acid method, in which the charring effect of the acid is apt to obscure the test when only minute quantities are present. We have satisfactorily used this test for four years, and find it delicate to one part formaldehyde in 300,000 parts milk.[b]

Mr. J. P. Street reported qualitative tests as follows:

To 620 cc of water were added 1.63 grams of formalin, equaling 1 part of formalin in 1,000. Ten cubic centimeters of this diluted formalin were added to 490 cc of milk, equaling 1 part of formalin in 50,000. A portion of this was still further diluted to 1 part in 100,000 and one part in 200,000.

Test 1.—Ten cubic centimeters milk were shaken with 5 cc hydrochloric acid, containing a drop of ferric chlorid and heated below boiling for three to five minutes. A violet color showed the presence of formalin.

Test 2.—Ten cubic centimeters of milk were taken and an equal volume of sulphuric acid added, containing a trace of ferric chlorid. The formalin was shown by the violet ring at the junction of the two liquids.

Test 3.—Five grams of coarsely powdered potassium sulphate were weighed into a 100 cc flask, 5 cc milk added, and 10 cc sulphuric acid carefully poured down the sides of the flask. A bright violet color was formed.

All of these tests gave a clear, characteristic reaction in each of the three diluted solutions. Test 1, though very apparent, gave a little weaker reaction than the other two tests in the solution, 1 to 200,000.

COPPER DETERMINATIONS.

Two reports on copper have been received, the one from Mr. R. W. Thatcher, of Washington, and the other from Mr. Charles A. Peters, of Idaho. Mr. Thatcher's work was planned "to show the effect of varying quantities and kinds of ammonium salts, or free ammonia, upon the end reaction when titrating with potassium cyanid," and his "observations seem to show conclusively that the use of sodium carbonate as the neutralizing agent, with a small amount of ammonium hydrate added to clear the solution and give the deep purple color, is the only reliable method of procedure." His methods were as follows:

Method I.—Dissolve in a measured quantity of ammonium hydrate (sp. gr. 0.96), and titrate directly.

Method II.—Dissolve in nitric acid, add measured excess ammonium hydrate, and titrate.

Method III.—Dissolve in hydrochloric acid, add measured excess ammonium hydrate, and titrate.

Method IV.—Dissolve in nitric acid, neutralize with sodium carbonate solution, add 1 cc ammonium hydrate (0.96), and titrate.

Method V.—Dissolve in hydrochloric acid—proceed as in IV.

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[a] Thirty-first An. Rep. Mass. State Board of Health, 1899, p. 606.

[b] The limit of delicacy was erroneously stated as 1 : 100,000 in our 1899 report.

Mr. Thatcher's results are as follows:

Determination of copper.

	Copper carbonate.			Paris green.		
	a.	b.	Average.	a.	b.	Average.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Method I	Not applicable.			22.28	22.34	22.31
Method II	56.00	56.14	56.07	24.32	24.00	24.16
Method III	53.88	53.64	53.76	23.97	23.63	23.80
Method IV	57.72	57.64	57.68	24.77	24.77	24.77
Method V	57.82	57.72	57.77	24.66	24.77	24.71
Moisture [a]	1.73	1.75	1.74	1.04	1.04	1.04

[a] Loss on drying eight hours at 105° to 110°.

Mr. Peters reports that Mr. H. T. Beans, who conducted the determinations, used the following procedure:

The paris green was dissolved in nitric acid, sodium hydroxid was added to the solution until a slight precipitate formed, 10 cc of a saturated solution of sodium carbonate were added, followed by 1 cc of ammonia (sp. gr. 0.90) the volume was made up to 100 cc, and the solution finally was titrated with potassium cyanid. The potassium cyanid was standardized against a copper-nitrate solution, the copper contents of which were determined electrolytically. The copper carbonate sent by referee contains considerable copper oxid. A portion of the copper carbonate furnished was ground, triturated, and the homogeneous sample thus obtained was used in this work.

Mr. Beans's results were as follows:

	Copper carbonate.			Paris green.		
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
Copper	55.56	55.63	55.60	24.81	24.59	24.70
Moisture	1.54	[a] 1.75	[a] 1.65	0.92	0.83	0.88

[a] These moisture determinations were made on the original sample of copper carbonate and not on the ground sample upon which the other determinations were made.

POTASSIUM CYANID.

Mr. C. L. Penny reports results with potassium cyanid, as follows:

Your letter of instruction on determining cyanogen, together with a sample of potassium cyanid has been received. I have to report the following results of analysis by the Liebig method, as stated by you. I weighed out 10 grams in duplicate from the center of the bottle, and made up to 1,000 cc. For each determination I used 50 cc equivalent to 0.5 grams. In one case the decinormal silver solution was 36.9 cc, in the other 37.0, the mean 36.95 cc. Calculating from the mean I find the cyanogen (CN) to be 38.49 per cent, and hydrocyanic acid (HCN) to be 39.97 per cent. A perfectly pure and water-free salt would call for CN=39.94 per cent.

The atomic weights used are K=39.15, CN=26.04, H=1.01. The method seems to me to be easy of execution, and to leave little to be desired. In a supplementary trial I added sodium chlorid, in amount equivalent to about one-half of the cyanid, and found the chlorid had no effect whatever on the result.

I tried chromate of potash as an indicator, and got 29.70 cc and 29.75 cc of silver for 0.2 gram of salt. This latter method gave 38.70 per cent cyanogen, as compared with 38.49 per cent by the Liebig method. The chromate method has the great disadvantage of showing chlorid as cyanid.

I should consider the Liebig method, as stated by you, admirable in every way, and leaving nothing to be desired.

Mr. Street, of the New Jersey Experiment Station, reported 39.29, 39.34, and 39.30 per cent of cyanogen in another sample of this material, using the same method. It would be well to state that the samples of potassium cyanid which were sent out were purchased already put up in 2-ounce bottles, and a difference in samples may account for this difference in results, which in view of this fact should not be considered so very great.

REMARKS.

Your referee on the subject of insecticides and fungicides regrets that his report can not be made of more interest by having the results of more work to report, and trusts that the associate referee, Mr. Haywood, will be able to counterbalance the deficiency by submitting a report of considerable work on the arsenicals.

The work here reported is so meager that your referee would refrain from making any recommendations based thereon. He would, however, venture to call the attention of the association to the following considerations:

First.—The detection and determination of formaldehyde does not properly belong to the referee on agricultural insecticides and fungicides. Except for fumigating closets, etc., or for use in the laboratory, I do not believe it is employed at all extensively.

Second.—The content of cyanogen in a sample of cyanid of potash is of interest, but for fumigating a greenhouse or similar purposes I believe a knowledge of the impurities and the character of the gases which they supply would be of greater value.

Third.—The purity of the lyes on the market for the home making of insecticide soap is of importance, but when it comes to the ready-made soaps themselves, there is much in the regular soap analysis that is of no interest to the entomologist.

From these considerations it would seem that much of minor importance might be eliminated from the work laid down by your referee which would result in lightening the labor of his successor. The recommendation is therefore made that the methods be continued with this in mind.

The President. We will now have the other portion of the report on insecticides.

Mr. Haywood. This is the report on arsenicals from the associate referee:

REPORT ON THE ARSENICAL INSECTICIDES.

By J. K. Haywood, *Associate Referee.*

At the request of Mr. Voorhees, the referee on insecticides, the associate referee took charge of the work on the arsenicals. Thirteen chemists signified their desire to take part in this branch of the work. The following directions were sent to each, accompanied by materials for analysis:

Dear Sir: You recently informed Professor Voorhees that you would collaborate in the work on insecticides. He has placed the work on arsenicals in my hands.

Inclosed you will find materials and methods for the determination of the two forms of arsenious acid in paris green. Will you kindly report all results as per cent and let me have them by October 15, if possible? Mix the sample of paris green well before analysis.

Very truly, yours,

J. K. Haywood, *Associate Referee.*

METHODS OF ANALYSIS FOR INSECTICIDES.

Paris Green.

MOISTURE.

Dry 1 to 2 grams for 8 to 10 hours at 105° to 110° C., and calculate loss as moisture. (In duplicate.)

TOTAL ARSENIOUS OXID.

Method I.

Solutions required:

Starch solution.—To prepare the starch solution, boil 2 grams of starch with 200 cc of water for about 5 minutes.

Iodin solution.—To prepare the standard iodin solution, dissolve 12.7 grams of powdered iodin in about 250 cc of water to which has added 18 to 25 grams of c. p. potassium iodid, and make the whole up to a volume of 1 liter. To standardize this solution weigh out 1 gram of the inclosed dry c. p. arsenious oxid; transfer to a 250 cc flask by means of about 100 cc of a solution containing 2 grams of sodium hydrate in each 100 cc, and boil until all arsenious oxid goes in solution; cool; make to a volume of 250 cc and use 50 cc for analysis.

This 50 cc portion is concentrated by boiling in a 250 cc flask to half its volume and allowed to cool to 80° C. An equal volume of concentrated hydrochloric acid is now added, accompanied by 3 grams of potassium iodid, mixed, and the whole allowed to stand for 10 minutes (to reduce the arsenic oxid, formed on boiling an alkaline arsenite, to arsenious oxid). The brown solution is then diluted with cold water and an approximately N/10 solution of sodium thiosulphate added, drop by drop, until the solution becomes exactly colorless. (This end point is easy to read without the aid of starch.) This solution is then made slightly alkaline with dry sodium carbonate (using a drop of methyl orange to read the change), and made slightly acid with hydrochloric acid, *taking care that all lumps of sodium carbonate on the bottom are acted on by the hydrochloric acid.* Sodium bicarbonate is now added in excess and the solution of iodin run in, drop by drop, using starch solution to read the end reaction. (Sometimes the solution gets dark toward the end of the titration. This must not be confused with the final dark blue color given by the iodin and starch.) From the number of cc of iodin solution used and the weight of arsenious oxid taken, the value of each cc of iodin in arsenious oxid can be determined.

Method (*Smith modified by Haywood*).—Two grams of paris green are weighed out and transferred to a 250 cc flask and about 100 cc of water and 2 grams of sodium hydrate added. This solution is boiled for 5 to 10 minutes, or until all of the green particles have changed to red cuprous oxid. It is then cooled to room temperature and the volume made to 250 cc. The well-shaken liquid is filtered through a dry filter and 50 cc taken for analysis. The analysis is carried out from this point forward the same as when we standardize the iodin solution. (In duplicate.)

Method II.

Solutions.—The solutions required are the same as above, with the addition of a solution containing 2 to 3 grams of sodium potassium tartrate in 50 cc of water.

Method (*Avery-Beans*).—Sample the paris green (as one would an ore for assaying) down to about 1 gram. Pulverize this small sample in an agate mortar and weigh out 0.2 to 0.3 gram in a beaker of, say, 300 cc capacity. Add 25 cc of water, and to the green suspended in the water add, with constant stirring, concentrated hydrochloric acid till solution is just effected. Six drops are usually sufficient. Now add to the acid solution sodium carbonate solution till a slight permanent precipitate is formed. Dissolve this precipitate by adding 2 to 3 grams of sodium potassium tartrate in solution. Now dilute to about 200 cc, add solid sodium bicarbonate and starch solution, and titrate with iodin in the usual way. (In duplicate.)

Method III.

(By precipitation with hydrogen sulphid and weighing as arsenious sulphid.) Dissolve with hydrochloric acid, filter, heat the solution in a flask to 70° C., and conduct hydrogen sulphid through as long as precipitation takes place. The precipitate formed is a mixture of sulphur, arsenious sulphid, and copper sulphid.

Spread out the filter in a porcelain dish with watch glass cover, and digest over water bath with sodium sulphid solution. Decant and repeat digestion two or three times. Finally, add some water, filter off with clear solution, and wash the residue with hydrogen sulphid water.

The sodium sulphid solution is warmed and made acid with hydrochloric acid, which reprecipitates the arsenious sulphid and a large amount of free sulphur; filter and wash. Extract the still moist precipitate on the filter with ammonia, wash the residual sulphur, reprecipitate the solution with hydrochloric acid without heat, filter in a Gooch crucible, dry, extract the remaining free sulphur with carbon disulphid, dry at 100° C., and weigh. Repeat extraction and drying until constant weight is secured. From the arsenious sulphid compute arsenious oxid. (In duplicate.)

Method IV.

(C. A. Goessmann.)

Weigh out 1 gram of the material in a beaker, add 50 cc of water and 5 grams of potassium hydroxid and digest on boiling water bath for half an hour. Filter and wash the precipitated suboxid of copper, make the filtrate acid with hydrochoric acid, and change the arsenious acid to arsenic acid by the addition of small quantities of chlorate of potash at frequent intervals and gentle heat. Drive off the excess of chlorin by repeated evaporations, make alkaline with ammonia, and precipitate with magnesia mixture. Let stand at least two hours, filter through a Gooch crucible, dry and burn, using in so doing the precautions mentioned in Fresenius. Cool and weigh as magnesium pyro-arsenate. (In duplicate.)

SOLUBLE ARSENIOUS OXID.

Method I.

Solutions.—A solution of starch and the standard iodin solution prepared as above; also a solution of sodium acetate containing 12.5 grams in each 25 cc.

Method—(*Avery-Beans*).—Digest over the flame 1 gram of paris green for about 5 minutes with 25 cc of a solution of sodium acetate containing 12.5 grams of the crystallized salt. The solution is then cooled, made up to 100 cc and 50 cc filtered off and titrated with standard iodin in the usual way. (In duplicate.)

Method II.

Solutions.—A solution of starch and the standard iodin solution prepared as above.

Method—(*Haywood*).—One-half gram of paris green is treated with 500 cc of water (previously boiled to get rid of CO_2 and again cooled to room temperature) in a large flask. The flask is stoppered and shaken 8 or 10 times each day for 10 days. At the end of this time the solution is filtered off through a dry filter. Two hundred cc of this are treated with sodium bicarbonate and titrated with iodin. This will give the percentage of arsenious oxid dissolved.

Another 200 cc of the solution is treated with 5 cc of concentrated hydrochloric acid, heated to 70° C., and a current of H_2S passed through until all arsenic and copper are precipitated. It is then filtered and washed quickly with hot water. The filter and its contents are burned with powdered sulphur and weighed in a porcelain crucible. In this way a mixture of CuO and Cu_2S is obtained. To calculate the percentage of CuO dissolved, consider the residue as entirely made up of CuO. This will be correct, since the per cent of copper in CuO and Cu_2S is the same. (In duplicate).

Please send both the figures for arsenious oxid and cuprous oxid dissolved by Method II and the reporter will calculate the CuO to arsenious oxid and subtract that amount from the total amount found.

Before going into the results obtained I will give a brief résumé of what has been done on the determination of soluble arsenious oxid in paris green, to at least explain why the methods as given above were chosen.

Up to about one year ago there had been no methods worked out for the determination of free arsenious oxid in paris green. Some chemists would allow the green to stand in contact with water for two to four hours and titrate the resulting solution with standard iodin; some would allow the green to stand in contact with water for twenty-four to forty-eight hours and titrate the resulting fluid; some would wash the green on a filter paper with a liter or two of water and titrate the resulting solution; some would extract the green with warm water, etc. By these methods many and diverse results were obtained on the same sample of paris green.

The first work that I can find on this subject was by Haywood and appeared in the Journal of the American Chemical Society in September, 1900. It was found during the course of the investigation there recorded that warm water could not be used to extract the soluble arsenious oxid since it broke up the paris green, also that it was not practicable to extract the soluble arsenious oxid by washing on a filter paper, since the wash liquor was still gaining in arsenious oxid, even after 2,000 cc had been used. It was also found that the soluble arsenious oxid went into solution much more slowly than had been known or anticipated, so that all had not gone into solution even at the end of a week, but seemed to require from ten to twelve days. A method was finally proposed based on these facts. It is as follows:

> Suspend one-half gram of paris green in 500 cc of water in a stoppered flask. Shake occasionally each day, and at the end of ten to twelve days filter off an aliquot portion and titrate with iodin solution for soluble arsenious oxid.

A month later an appendix appeared to this paper, in which the author further stated that copper oxid also went into solution to some extent during the extraction with cold water. The author was inclined to think that this was due to a breaking up of the paris green, and so assumed (no work having been done to prove the contrary) that the copper oxid and arsenious oxid went into solution in water in the same proportion in which they were present in the paris green, and consequently a nearer figure to the true per cent of soluble arsenious oxid could be obtained by correcting the total quantity by an amount corresponding to the copper oxid in solution.

During the same month, October, 1900, another paper on the soluble arsenious oxid in paris green, by Hilgard, appeared in the Journal of the American Chemical Society. With the former writer he agreed that warm water could not be used to extract the soluble arsenious oxid from paris green, but, unlike the former worker, he seemed to think that the copper in solution in the water came from verdigris, perhaps already present in the green, and that, after washing the green for a while, although arsenious oxid still came through in the wash water, copper oxid failed to do so, thus showing that the paris green did not break up when in contact with water, and that, consequently, a correction could not be applied to the soluble arsenious oxid based on the amount of copper oxid found in solution. He advised extracting 1 gram with 1,000 cc of water for twenty-four hours, at least, and using the resulting figure, obtained after titration, for free arsenious oxid.

In February, 1901, a paper by Avery and Beans appeared in the Journal of the American Chemical Society on this subject. They found, during the course of their investigation, that water does break up paris green to a great extent (depending on the fineness of the material), but that the amounts of copper and arsenious oxid going into solution are not in the same proportion as that in which these substances are present in the original green. Although they were of the opinion that approximate results could be obtained by suspending 1 gram of paris green in 1,000 cc of water for a week, they said that this method was purely arbitrary, and therefore advocated extracting the paris green with a boiling solution of sodium acetate, which compound not only renders the free arsenious oxid present more soluble than it would be in pure water, but retards the dissociation of the paris green, thus causing it to be broken up to a much less extent. They also found that water saturated with CO_2 dissolved about one-half the arsenious oxid from a sample of paris green during the course of a week.

In this connection it may be of interest to show some figures that we have obtained at the Bureau of Chemistry, Department of Agriculture, comparing the water extraction and sodium acetate extraction methods, and to call attention to certain facts brought out by them.

Extraction of arsenious oxid by water and by sodium acetate.

Number.	Arsenious oxid soluble in water.	Copper oxid soluble in water.	Arsenious oxid soluble in sodium acetate solution.	Number.	Arsenious oxid soluble in water.	Copper oxid soluble in water.	Arsenious oxid soluble in sodium acetate solution.
	Per cent.	*Per cent.*	*Per cent.*		*Per cent.*	*Per cent.*	*Per cent.*
1	9.30	1.90	.88	1	2.16	.90	1.08
2	8.81	1.45	1.17	2	4.11	.65	1.17
3	6.37	.65	1.76	3	4.32	.55	1.17
4	6.97	1.65	1.08	4	4.41	1.05	1.08
5	5.52	.95	1.17	5	5.39	.90	.88
6	10.53	.60	8.98	6	2.93	....	.88

From this table it will be seen that the figures for arsenious oxid by water extraction are much higher than those by sodium acetate extraction, even when the water extraction figures are corrected by subtracting an amount of arsenious oxid corresponding to the amount of copper oxid in solution. But we would like to call your attention to another fact. The first six of these greens are the very coarsest that could be found among a group of forty-seven and the second six the finest. It therefore appears that the water extracted in every case more arsenious oxid from coarse paris greens than from the fine ones; yet Avery and Beans found that the finer the green the more arsenious oxid extracted in a given length of time. We agree with Avery and Beans that water does break up paris green, and that for any certain green the finer the green the more it will be broken up by water in a given length of time, since more surface is exposed to the action of the water. Yet these figures would seem to show the contrary. This apparent discrepancy is, perhaps, explained when we consider the following fact. The coarser paris greens are considered in most cases not to be made as well as the finer ones. Might it not be the case, then, that such greens have not been boiled long enough in acetic acid, and that a certain part of them is in a very loose combination, which combination is readily broken up on treatment with water? On the other hand, when such samples of paris green are treated with sodium acetate we would have the following conditions. Paris green would be suspended in a solution containing a large amount of sodium acetate, which, as is well known, is hydrolyzed to a great extent in water solution according to the following scheme:

$$NaCH_3COO + HOH \rightleftarrows NaOH + CH_3COOH.$$

Might not the acetic acid act on the sample in question and cause the arsenious oxid to form a more stable compound, so that nearly all of the arsenious oxid going into solution would be that not combined in any way with the other constituents?

Since a small amount of NaOH also exists in the above solution we would naturally expect that it would act on the paris green to some extent, and set a little arsenious acid free over and above that present in the free state, along with some copper. Such an action very likely does take place, since small amounts of copper are always found present in solution on extracting the green with sodium acetate. That such an action would be small as compared with the "combining" action of the acetic acid in solution seems to be borne out by the above figures.

Looking at the matter in this light, it would seem that even though water extraction does not give the exact amount of free arsenious oxid in the green, it at least gives us some idea of its stability and is consequently of value.

For these reasons your associate referee has chosen the sodium acetate method as the best one now at our command to determine the actual soluble arsenious oxid in paris green, and the water extraction method as the one which shows to some extent the stability of the green in question and gives some idea of what its action would be in actual orchard practice.

Following are the results obtained by five chemists. The other eight were not able, because of press of work, to send in any figures at all.

Estimation of total and soluble arsenic.

Analyst.	Moisture.	Total arsenious oxid.				Soluble arsenious oxid.		Soluble copper oxid.
		Method I.	Method II.	Method III.	Method IV.	Method I.	Method II.	Method II.
	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
H. T. Beans, Moscow, Idaho	0.92	56.29	56.63	....	....	0.78	0.86	0.30
	.83	56.43	56.71	....	....	.86	.96	.40
	....	....	56.63	....	....	....	....	....
R. W. Thatcher, Pullman, Wash	1.04	56.12	56.16	....	....	.55	2.94	....
	1.04	56.03	56.29	....	....	.52	2.88	....
J. K. Haywood, Washington, D. C	.91	56.68	56.84	....	[a] 56.10	.78	2.08	.40
	.88	56.80	56.82	....	56.00	.81	2.69	.55
	....	56.80	....	....	....	....	....	....
G. W. Cavanaugh, Ithaca, N. Y	....	55.88	57.16	....	[a] 56.30	....	....	....
	....	56.00	57.16	....	56.35	....	....	....
	....	56.13	....	....	....	....	....	....
H. B. Slade, Lincoln, Nebr	Trace.	56.69	55.98	....	56.10	.42	....	....
	....	56.81	56.31	....	55.97	.42	....	....

[a] Corrected as directed by Fresenius for the solubility of the magnesium ammonium arsenate precipitate in the mother liquor.

REMARKS.

H. T. Beans.—(1) In Method IV for total arsenious oxid considerable amounts of iron from the paris green and silica from the glassware interfered with the determination.

(2) An objection is made to the standardization of the iodin solution for the tartrate method (Avery-Beans) by the process outlined for the Smith-Haywood method, unless it can be shown that there is an appreciable oxidation of arsenic during its solution as carried out by the ordinary method of preparing alkaline arsenite solution. The authors (Avery-Beans) prefer to standardize the iodin solution in the regular way, i. e., by the use of a solution containing 4.95 grams of arsenious oxid in a liter of water.

R. W. Thatcher.—I did not attempt the estimation of arsenic as sulphid, as my laboratory is not equipped for combustion in hydrogen, and furthermore I believe that the method is practically obsolete as far as being a practical method for technical use. I did attempt the determination by the Goessmann method, but upon making the solution alkaline for precipitation with magnesia mixture a very perceptible quantity of aluminum hydroxid appeared, probably derived from the dish in which the paris green was digested with caustic alkali. Furthermore, the solution to which the magnesia mixture was added gave a continued deposition of crystalline matter for several days. I finally became disgusted and threw away the whole of the precipitated matter. I am very strongly of the opinion that the gravimetric methods as at present outlined are entirely worthless.

J. K. Haywood.—I was not able to get reliable results by Method III for total arsenious oxid. Nearly always the copper sulphid would run through the filter after being separated from the arsenious sulphid by sodium sulphid. Even when this did not happen the final results on arsenious sulphid were very variable.

Geo. W. Cavanaugh.—I omitted Method III for total arsenious oxid, since from previous experience I found that I could not get reliable results with it.

H. B. Slade.—The determination of arsenic as arsenious sulphid was made, but the results were so divergent, owing to the large amount of sulphur present, and the time taken in washing out the sulphur led me to omit the completion of this method. The Smith-Haywood method (I) for total arsenic apparently gives high results from lack of sharp end reaction with thio-sulphate in the preliminary treatment. The iodin is thus titrated against it and the percentage increased by this error. The same error, if present, is involved in the standardization of the iodin solution. In each case in the determinations reported 19.5 cc of thio-sulphate solution were necessary to take up the iodin set free in this method. The error, then, appears constant.

RECOMMENDATIONS.

Your associate referee would recommend that Methods I and II be adopted provisionally and tested again next year, and that both methods for soluble arsenious acid be given another trial.

The PRESIDENT. Are there any special papers to be presented on the subject relating to the analyses of insecticides? Are there any remarks or is there any discussion of the report?

Mr. ALWOOD. I would like to know whether there is any method of treating paris green so as to get rid of the soluble arsenious acid and to prevent the paris green itself from breaking up? Is there any method by which the horticulturist or farmer could treat the paris green?

Mr. HAYWOOD. It has been thought for a long time that if paris greens were treated with calcium hydroxid in solution it would render the free arsenic more insoluble, or at any rate improve them. Personally I believe that calcium hydroxid may break up the paris green to some extent, and really renders the total arsenic more soluble, but that this arsenic is not more soluble as arsenious acid but as calcium arsenite, which does not seem to injure the foliage as much as arsenious acid. Probably more work will have to be done on this before we can tell what the action of calcium hydroxid is.

Mr. ALWOOD. Some years ago we did some work in our laboratory with reference to the soluble arsenic in London purple, and, whatever the explanation was, it was very evident that the addition of quicklime or calcium hydroxid to the London purple diminished very noticeably—not entirely, but below the danger point—the solubility of the arsenic.

(The president asked Mr. Wheeler to take the chair, and then spoke from the floor.)

Mr. VAN SLYKE. I want to make just one or two statements in regard to the subject of soluble arsenic in water. Now it seems to me that it is desirable, in determining the amount of soluble material in paris green, to keep in mind the specific reason for which we want to make the determination. It is known that arsenic in soluble form injures foliage when it is present beyond certain quantities, and it seems to me that in our methods we need to keep in mind this particular thing. This is a question of scientific interest, and it is desirable to determine what the specific effect of sodium acetate is and what the specific effect of the water is on the paris green. But when it comes to the practical application of our results in horticultural work, it seems to me that we ought to adopt the method which will apply to the question of the determination of the danger point in the application of the arsenic to foliage. In my own judgment it appears that we should approximate somewhat the conditions which maintain in horticultural operations. Ordinarily the paris green is mixed with the water not very long before it is applied, probably not more than a few hours at the most. It is possible, but I do not think it is a common custom, for horticulturists to mix the paris green and let it stand indefinitely before using. Now, if the paris green is in contact with the water only a few minutes or a few hours before application, what is the use of making a determination of the amount of arsenic that is soluble in sodium acetate or in water on standing a week, more or less? In the absence of any better guide in our own laboratory, where we are obliged to look after the paris green of the State by way of inspection, I have provisionally adopted the method of putting the water on paris green at the rate of 10 parts of water to 1 of paris green and shaking occasionally and letting it stand in contact for twenty-four hours. Now, it is safe to say that if that treatment fails to develop an amount of soluble arsenic that will be sufficient, in the opinion of horticulturists, to injure the foliage, then it is perfectly safe to pass that paris green as all right. In working over the samples that are present in the market of New York State, I believe there was only one that failed to respond to this test; that is, there was only one sample that appeared to contain sufficient soluble arsenic to make it possible that the foliage might be injured. On the other hand, where we let it stand ten days or two weeks the amount of soluble arsenic was so large that scarcely any of the samples were of a kind to be permitted in horticultural operations. So I say that it seems to me that while it is desirable to study the actual chemical changes that take place, we want to keep in mind the application, and that we want, if possible, to approximate conditions in our methods that will enable us as nearly as possible to tell how much soluble arsenic is present in the paris green that in the hands of the horticulturist, under the methods used by him, is likely to do harm.

Mr. Haywood. I would like to say in this connection that in determining the soluble arsenic in paris green, although you may get certain results by dissolving for twenty-four hours, or overnight, really the result which gives you more closely the effect the paris green would have in actual orchard practice is to let it stay a good length of time in contact with water, then you will have some idea of its stability. It depends a great deal on the stability of the paris green, it appears to me, whether it will scorch the foliage or not, because an unstable sample of paris green, under adverse conditions, with a great deal of moisture and perhaps quite a little carbon dioxid coming in contact with it, might scorch the foliage. So it appears to me that what really is needed is to understand paris green from the standpoint of its stability even more than from its sodium-acetate-extraction standpoint. Even the free arsenic that is present hardly begins to pass into solution in twenty-four hours, leaving out of consideration the breaking up of the paris green.

Mr. Van Slyke. I would like to ask Mr. Haywood if he has any information which shows whether, after the paris green is applied to the foliage, any continuous change goes on? I have not been able to find any information published on that point.

Mr. Haywood. I have not been able to find any information on that point. I can simply repeat what Avery and Beans said in their article recently—that they not only applied water to the paris green, but they applied water saturated with carbon dioxid, and they found that the water gradually broke up the paris green, more so in some cases than in others; also that when the water was kept saturated with carbon dioxid inside of a week a large part of the paris green had gone into solution; in other words, the arsenic had been rendered soluble. And they found out that if they ground up the green rather fine, at the end of a week they would have a very large amount of the arsenic going into solution.

Mr. Alwood. Let me say that this is the point of my first question. Paris green often scorches the foliage. In a wet season this becomes worse; undoubtedly there is carbon dioxid in the rain water and in the dew, and the solution of the arsenic goes on until the tree may be quite defoliated under some conditions, while under other conditions it will be quite safe. We apply arsenites in large quantities, and it is of vast importance to fruit growers. We use lime constantly, and we mix it with the poison just before we apply it.

Mr. Van Slyke. Of course it is known to be true in actual practice that paris green is less injurious when used with lime.

Mr. Alwood. We noticed that.

Mr. Haywood. When I spoke just now I did not mean to say that lime did not cause the paris green to be less hurtful. It most certainly does. I simply advanced the proposition, more as a theory than anything else, that paris green is broken up to some extent by lime, and that the calcium salt of arsenious acid or calcium arsenite, although quite soluble in water and greater in amount than the original free arsenic, does not hurt the foliage as much as the small amount of free arsenious acid itself.

The Chairman (Mr. Wheeler). We will proceed to the consideration of the next subject in order, the report on dairy products, which will be read by Mr. Van Slyke.

Mr. VAN SLYKE. The report was prepared by the referee, Mr. Le Clerc, in the laboratory at Geneva, and as he has gone abroad since preparing the report, and as Mr. Cavanaugh, the associate referee, who expected to read the report, is absent, I have taken the responsibility of presenting it.

REPORT ON DAIRY PRODUCTS.

By J. A. LE CLERC, *Referee.*

The referee this year divided his work into two divisions:

I. Determination of albumin in milk.

II. Detection of renovated butter.

I. DETERMINATION OF ALBUMIN IN MILK

The work on casein in milk has been done so satisfactorily by former referees that the methods of determination of casein as adopted by this association leave nothing to be desired. Not so, however, with the determination of albumin. This determination in the hands of different chemists has never given concordant results. Hence, it was decided to confine our work, in so far as milk was concerned, to the determination of albumin.

Letters asking for cooperation were sent to eighteen chemists. Fourteen replies were received, eleven of which were from men who signified their willingness to cooperate.

On June 21 samples of skim milk, preserved with formalin, were sent to seven men, with the following instructions:

I. (*a*)[a] Remove casein by the official method, Bulletin 46, Revised Edition, p. 55, par. 1.

(*b*) Albumin, provisional method: Use filtrate of (*a*), neutralize exactly with potassium hydroxid, and add 0.3 cc 10 per cent acetic acid; heat in water bath till separation of albumin is complete and supernatant liquid is clear. This may require one hour or more. Determine nitrogen in the precipitate.

II. (*a*)[a] Remove casein by precipitation with alum as follows: To 10 grams milk and 50 cc water, at 45° to 50° C., add 1.5 to 2 cc saturated alum solution, filter, and wash.

(*b*) Albumin in filtrate of (*a*): Heat in water bath (without neutralization or addition of acid) till precipitate is complete and supernatant liquid is clear. Determine nitrogen in the precipitate.

(*c*) Albumin in the filtrate of (*a*): Neutralize filtrate of (*a*) with potassium hydroxid. Filter off any precipitate of alum hydrate that may be formed. If no precipitation takes place add a few drops of alum solution. Filter precipitate. In either case wash precipitate thoroughly and to the filtrate add 0.3 cc 10 per cent acetic acid. Heat in water bath till supernatant liquid is clear after the precipitation. Determine nitrogen in the precipitate.

III. (*a*) Penny's method: Place 50 gm milk in 500 cc flask, add 250 to 300 cc water at 45 to 50° C. Add 10 to 12 cc saturated alum solution till casein is completely precipitated. Fill to the mark. Filter.

(*b*) Heat 100 cc filtrate of (*a*) as in II (*b*). Determine nitrogen in albumin precipitate.

(*c*) Treat 100 cc filtrate of (*a*) as in II (*c*). Determine nitrogen in albumin precipitate.

(*d*) Determine nitrogen in filtrate of (*c*), which equals nitrogen other than casein and albumin nitrogen.

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[a] Casein may be determined quantitatively if you have time, in which case determine also the total nitrogen in the milk.

On July 25 another sample of skim milk was sent to four other chemists. The work on albumin by the association began in 1894, when Dr. Van Slyke read a paper on the determination of albumin in milk. The method consisted in heating directly the filtrate of casein (which has been precipitated by acetic acid) about fifteen minutes in water bath. The results always showed that there were other bodies in milk besides casein and albumin, bodies which were neither precipitated by acetic acid nor coagulated on heating the filtrate. These bodies amount in many cases to 10 to 12 per cent of the total protein.

In 1896 the above method was recommended for adoption by the association.

In 1897 the association made a study of the determination of albumin wherein, besides the above method, the acetic-acid filtrate was neutralized (after the precipitation of casein), and then there was added 0.3 cc 10 per cent acetic acid, and the solution then heated on the water bath till albumin was completely precipitated. Results were obtained with the latter modification only from the New York (Geneva) Station, and showed a slightly higher amount of albumin than was obtained by boiling the acetic-acid filtrate direct. It was thought that the large amount of acetic acid might keep some of the albumin in solution, hence, the need of neutralization and the addition of only enough acid to acidify distinctly.

In 1898 the association's work on albumin in milk was changed. Strong solution of tannin was added to the filtrate from casein. Results showed a higher amount of albumin than ordinarily occurs in milk. This is due to the fact that tannin throws down albumoses and peptones, bodies which may be present in milk that is a day old or more.

In 1899 the association continued work with tannin and also tried another method. This was to precipitate the casein by means of alum and then to determine the total nitrogen in the filtrate and call that nitrogen albumin. Obviously, the results were high, because all the noncasein nitrogen can not be assumed to be albumin, unless, perhaps, in fresh milk, and even then the assumption could hardly be absolutely true.

Last year (1900) the association continued the work on the determination of albumin as in the two previous years, but presented one new method. This consisted in precipitating the casein by magnesium sulphate and determining the albumin in the filtrate after the addition of 0.3 cc 10 per cent acetic acid and boiling. This latter method agreed quite closely with the determination of albumin in the filtrate from acetic acid, which was neutralized, 0.3 cc 10 per cent acetic acid, and boiled. By the other methods, where tannin was employed as a precipitant, much lighter results were obtained. In that year it was recommended to adopt the use of tannin as a precipitant for albumin. This recommendation was not adopted, however.

Though the results this year are not so concordant as was hoped for, yet, if the use of tannin as a precipitant for albumin is shown to be erroneous, something will have been gained. This year it was proposed to obtain albumin only by means of heat. I believe, so far as we now know, there is no distinctly albumin precipitant. Heat seems to be the only safe means of precipitating or rather coagulating albumin. Neither albumoses nor peptones nor amid bodies are affected by heat, while both albumoses and peptones are precipitated by tannin.

The proposed methods for the determination of albumin were—

(1). Neutralize the acetic-acid filtrate (after the removal of the casein by the official method) with potassium hydroxid, adding 0.3 cc 10 per cent acetic acid and heat on the water bath till supernatant liquid is clear. (2) Remove casein by use of alum, and coagulate albumin in the filtrate by heat (a) directly, (b) after removal of excess of alum by precipitating it with potassium hydroxid, filtering, and making acid with 0.3 cc acetic acid and heating. (3) Repeat (2) with the filtrate obtained by Penny's method.

It has also been the referee's desire to show conclusively that there is a large amount of nitrogen left in the milk after the casein and albumin have been removed, and, to accomplish that, an extra determination was made in the filtrate from albumin.

Following are the results obtained in Sample No. 1.

Determination of albumin in milk, No. 1.

Analyst.	Total protein.	Casein.			Albumin.					Protein other than casein and albumin.
		I (a).	II (a).	III (a).	I (b).	II (b).	II (c).	III (b).	III (c).	III (d).
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
A. J. Patten, Geneva, N. Y.	3.41	2.69 2.68	2.66 2.69 2.68	2.71	0.325 .319	0.240	....	0.270 .300	.... No precipitate.	....
J. A. Le Clerc, Geneva, N. Y.	3.47	2.79	2.78 2.78	2.66	.262 .275	.287 .280 .319	Slight precipitate.	.287 .312	.312 .312	0.301 .319
C. L. Penny, Newark, Del.	3.42	....	....	2.69	....	.319	....	....	....	[a].737
A. T. Charron, Ottawa, Canada	....	....	....	....	[a].173	.345	.375	.372	.328	.291
C. H. Jones, Burlington, Vt.	3.57	2.79	2.75	....	.270	.250	.270	.210	.290	.460
		2.79	2.79	....	.250	.270	.290	.210	.310	.540
		2.78	2.75	....	.250	.280	.280	.210	.280	....
A. L. Knisely, Corvallis, Oreg.	3.41	2.76	2.77	....	.240	[a].090	[a].130	[a].160	[a].030	[a].170
		2.78	2.77	....	.240	.200	.140	.160	.020	.210
		2.81	2.83	....	.210	.170	.100	.130	.070	.240
Highest	....	2.81	2.83	2.71	.328	.345	.375	.312	.312	.540
Lowest	....	2.68	2.68	2.66	.210	.240	.270	.210	.280	.291
Average	....	2.76	2.75	2.69	.265	.290	.304	.260	.305	.382

[a] Omitted from averages.

It will be seen that I (b), II (b), and III (b) give practically identical results, the averages being 0.265, 0.29, and 0.26 per cent albumin, respectively.

The results where the alum was precipitated and filtered are not at all satisfactory. In some cases the alum hydrate appears to retain mechanically nitrogen compounds that are difficult to remove by washing.

The referee made a complete analysis of milk No. 1, using alum to precipitate casein, heating the filtrate directly for albumin, acidifying filtrate with hydrochloric acid and precipitating the albumoses and peptones by means of bromin, and determining the amids in the filtrate from bromin. Ammonia was determined in the milk directly by distillation with magnesium oxid.

Complete analysis of the nitrogenous bodies in milk.

	Per cent.	Per cent of total protein.
Casein	2.78	79.7
Albumin	.28	8.0
Albumose and peptones	.21	6.0
Amids	.21	6.0
Ammonia	.01	.30
Total	3.49	100.00

Work on the proteolysis of milk at the Geneva station shows that whereas the casein constitutes over 80 per cent of the protein in fresh milk 50 per cent of this casein becomes, under certain conditions, converted into albumoses, peptones, and amid bodies.

It would seem necessary, therefore, that our methods for the determination of casein and albumin should give us casein and albumin. It is time that this association should make a more systematic study of the nitrogen compounds of milk. Surely, if we go to a great deal of trouble to determine the albumin, which constitutes from 6 to 8 per cent of the protein, we should give some attention to the determination of the other bodies constituting over 10 per cent of the protein of the milk.

Sample No. 2 was sent out July 25 to six chemists. The only results obtained were from the Geneva station.

This sample of skim milk had been heated, thus reducing below normal the amount of albumin present. However, it answered the purpose of showing that there was almost twice as much noncasein nonalbumin nitrogen as there was albumin, and any method of obtaining the albumin by using tannin as a precipitant, or of calling all the noncasein nitrogen albumin, would give excessively high and erroneous results.

Determination of albumin in milk, No. 2.

Analyst.	Total protein.	Casein.			Albumin.					Protein other than casein and albumin.
		I (a).	II (a).	III (a).	I (b).	II (b).	II (c).	III (b).	III (c).	III (d).
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
A. J. Patten, Geneva, N. Y.	2.72	2.28	2.26	2.24	0.135 .151	0.151 .158	0.092	0.158 .151	0.101	0.246
J. A. Le Clerc, Geneva, N. Y.	2.65	2.26	2.28	2.24	.115 .125	.140	.099 .107	.158 .158	.101 .118	.209 .242
G. E. Patrick, Washington, D. C.[a]	2.69 2.69	2.19 2.00	2.13 2.06 2.12	....	.04 .07	.13 .11 .16	.07 .09 .07	.16	.13 .14	.31
Highest	2.72	2.28	2.28	2.24	.151	.158	.107	.158	.118	.246
Lowest	2.65	2.26	2.26	2.24	.115	.140	.092	.151	.101	.209
Average	2.69	2.27	2.27	2.24	.132	.149	.099	.156	.107	.232

[a] Received too late to be included in the average.

Report on official milk work.[a]

[By T. C. Trescot and D. Stuart.]

	Per cent N.	Per cent, N×6.25.	Mean.
Total amount in sample	0.43	2.69	2.69
	.43	2.69	
I. (*a*) Casein	.35	2.19	2.10
	.32	2.00	
(*b*) Albumin	.006	.04	.06
	.011	.07	
II. (*a*) Casein	.34	2.13	2.10
	.33	2.06	
	.339	2.12	
(*b*) Albumin	.020	.13	.13
	.017	.11	
	.025	.16	
(*c*) Albumin	.011	.07	.08
	.014	.09	
	.011	.07	
III. (*b*) Albumin	.025	.16	.16
(*c*) Albumin	.020	.13	.135
	.022	.14	
(*d*) Nitrogen other than casein or albumin	.05	.31	.31
Other proteids from I (*b*)	.060	.37	....
Other proteids from II (*b*)	.062	.39	....
Other proteids from III (*b*)	.062	.39	....

[a] Work done September 25 to October 19, 1901.

II. DETECTION OF RENOVATED BUTTER.

On June 20 the referee sent out samples of renovated and dairy butters, marked simply "A" and "B", to six chemists, with instructions relating to the detection of renovated butter. Only three men sent in results. Most of the methods proposed were those suggested by Messrs. Hess and Doolittle, published in the Journal American Chemical Society, March, 1900. The microscopical test was that of Mr. Hummel, to be found in the same journal for June, 1900.

The following are the methods as sent out for comparative trial.

(1) Try the curd test with both samples A and B. (Journal American Chemical Society, March, 1900, p. 151.)

(2) Appearance of the curd. (Ibid.)

(3) Identification of the source of the curd in A and B. (Ibid.)

(4) Quantitative estimation of the curd (A and B). (Ibid.)

(5) Microscopic test of the curd with polarizing microscope, using selenite plate. (Journal American Chemical Society, June, 1900, p. 328; Bulletin 46, revised edition, p. 54 (m).)

(6) Shake 3 to 4 grams A, in test tube, with ether. Pour off fat (dissolved in the ether) into evaporating dish. Do the same with B. Let evaporate spontaneously twenty-four hours or more and compare appearance of residue.

Results of tests for renovated butter.

Test No.	Analyst.	Sample A (renovated butter).	Sample B (dairy butter).
1	A. J. Patten, Geneva, N. Y.	Sputtered	Foamed.
	A. M. Peter and J. O. La Bach, Lexington, Ky.	No foam; acted like oleo	Foamed freely, like genuine butter.
	R. W. Thatcher, Pullman, Wash.	No foam; sputtered vigorously	Foamed freely.
2	A. J. Patten	Curd separated slowly; melted fat had cloudy appearance.	Melted fat clear.
	A. M. Peter and J. O. La Bach.	Appearance of curd same in both cases; curd granular.	
	R. W. Thatcher	Curd settles slowly and granular, with small lumps.	Melted fat clears quickly; curd smooth and gelatinous.
3	A. J. Patten	Test not satisfactory.	
	A. M. Peter and J. O. La Bach.	Test abandoned.	
	R. W. Thatcher	Filtrate from fat gives flocculent precipitate when boiled with acetic acid.	Filtrate boiled with acetic acid gives slight opalescence.
4	A. J. Patten	Test not satisfactory.	
	A. M. Peter and J. O. La Bach.	Test abandoned.	
	R. W. Thatcher	Per cent casein, 0.54; per cent albumin, 0.062; ratio, 8.78 : 1.	Per cent casein, 0.262; per cent albumin, 0.012; ratio, 21.7 : 1.
5	A. J. Patten	Yellowish-green field mottled with blue, very distinctive.	Uniformly blue field.
	A. M. Peter and J. O. La Bach.	Crystals in both samples, but most characteristic in A.	
	R. W. Thatcher	Blotches of semicrystalline matter visible.	Smooth, clear field.
6	A. J. Patten	Remained clear yellow	Colorless after standing 3 to 4 days.
	R. W. Thatcher	No difference in the curd.	

It will be noticed that the tests are quite characteristic, notably the foam test, and the microscopic test. When samples A and B were sent from Geneva, both samples were in good condition, and could not be distinguished one from the other by any superficial test.

Mr. Patten makes the following comments:

(1) A melted quietly and clear, but on being heated longer, began to sputter, and remained a clear yellow on cooling. B melted with more or less frothing, and became darker in color on cooling.

(2) B on being melted gave a clear supernatant liquid, while with A the curd separated very slowly, giving the melted fat a cloudy appearance. The difference in appearance of the curd was not so distinct, and would not be reliable.

(3) B gave a uniformly colored yellowish-green field. A gave a yellowish-green field mottled with blue, very distinctive.

(4) B became colorless after standing three to four days, while A remained a clear yellow. On dissolving A in ether in the test tube a whitish semifluid substance was left adhering to the sides after pouring off the ether, which substance did not appear when B was treated in the same way.

A. M. Peter and J. O. La Bach say "these results are not very satisfactory, but they appear to show that A is process butter."

R. W. Thatcher comments as follows:

In (3) the temperature at which fat is melted must be kept below 80° C., or the albumin is coagulated, and does not appear in the filtrate.

(5) Gives a very characteristic distinction, if the butter is kept well cooled before and during the examination.

Recorded results under (4) are those of a single determination, which did not proceed very satisfactorily, hence should not be relied on too greatly.

Hess and Doolittle say that "if the artificial curd of process butter has been derived from milk, then the ratio of the percentage of casein to the percentage of albumin should be the same as that ratio is in milk, or about 9 parts casein to 1 part of albumin." Mr. Thatcher actually found the ratio to be 8.78 : 1, but, as he says, this is not to be relied upon too greatly.

It was the referee's intention, when the results were all in and comments noted, again to send out samples, this time using only those methods which appeared to give characteristic results, and also to give trial to any suggestions which might be offered by the coworkers.

Farmers' Bulletin 131, by G. E. Patrick, confirms the foam test, which has been successfully used for the past ten years for the detection of oleo.

The above results will only show that the sample under examination is or is not genuine butter; but if it is not genuine butter, the ordinary chemical methods for the examination of the fat will at once show whether the sample be oleo or renovated butter.

RECOMMENDATIONS.

First. Continued work on determination of albumin in milk is suggested.

Second. Continued work on adulterations of butter is advised.

President Van Slyke resumed the chair.

The President. Mr. Patrick began some work on the subject of renovated butters some time before it was taken up by the referee, and his results cover the ground more thoroughly, probably, than the report of the referee, and he will, in accordance with my request, give his paper separately.

Mr. Patrick. But as a part of the referee's report. It is supposed to be handed in to him, but it was too late in finishing.

Mr. Street. I have a resolution that I would like to offer, if in order, and ask that it be referred to the committee on resolutions for favorable report:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

Mr. Leach. Mr. President, may I say a word with regard to the systematic examination of preservatives for milk? It seems to me that the subject is one that we ought to consider carefully, in view of the marked increase of late in this form of adulteration. In the Massachusetts Board of Health laboratory we examine milk collected from all parts of the State, with the exception of Boston, which has a very efficient system of its own. During the summer months, in addition to our regular analysis of milk for solids and fat, we examine for preservatives every sample of milk that is brought in, amounting to some 500 samples per month, and varying from 20 to 60 per day. This is not as difficult as at first sight appears, if reduced to such a system as I have adopted.

I might say at the outset that in 1900, for instance, from June to September, inclusive, upward of 2,000 samples were examined, of which about 3 per cent contained formaldehyde, approximately one-half of 1 per cent contained boracic acid, and a very few samples contained carbonate of soda. My method of procedure is as follows:

The milk samples are arranged in the order of their numbers, and about 10 cc of each is poured into a casserole, the casseroles being arranged in the same order as the containers of the samples. Each casserole is then treated alike in succession, first adding from a large stock bottle fitted with a siphon about 10 cc of concentrated hydrochloric acid containing a very little ferric chlorid, then holding the casserole over the gas flame and giving it a rotary motion while heating. The well-known violet coloration, indicative of formaldehyde, will appear if as much as 1 part in 300,000 is present. Less than one minute is required in making this test on each sample.

I am aware that this method has been the subject of some criticism, due doubtless to the fact that various aldehydes when present in milk give color reactions under this treatment. I have, however, found nothing but formaldehyde that would give the peculiar violet color, which is very distinctive. I have tested its reliability again and again during the last five years, and while using it only as a preliminary test, I may say that I have always been able to confirm it.

The tests for boracic acid and carbonates are made incidental, as it were, to the cleaning of the platinum dishes in which the determinations of total solids were made, so that very little extra time is involved. The residues are first incinerated in the original numbered dishes, which are afterwards arranged in order on a tray. With a pipette, one or two drops of dilute hydrochloric acid are added to each ash in succession, noting effervescence, which, of course, indicates carbonate, to be confirmed later by the rosolic acid test.

By means of a wash bottle a few centimeters of water are added to each acidulated milk ash, the tray is given a rotary motion to aid in dissolving the ash, and a strip of turmeric paper is first immersed in each solution and then allowed to dry while adhering to the side of the dish, out of contact with the liquid. The usual cherry-red color on the paper when dry indicates boracic acid.

Thus the three preservatives most commonly used are very readily singled out, confirmatory tests to be afterwards made, of course, on fresh samples of the milk.

The PRESIDENT. I do not know but that Mr. Patrick's paper had better be put over until after recess, when there will be no danger of interruption. If there is no objection, we will have this paper the first thing this afternoon.

Mr. PATRICK. Before we leave the subject of analysis of milk I would like to suggest that perhaps the different results, of which the president spoke, obtained by the chemists, in the determination of albumin and casein, might even in the case of those preserved samples have been due to enzyms—to changes produced by the enzyms. I have not seen the figures, but the reason I suspected this was, that in our laboratory, in the work being done by Mr. Stuart and Mr. Trescot, the figure for albumin became exceedingly low, about 0.13 per cent. I presume that that is the low figure, or one of them, that the referee referred to. Those analyses were not made until a few weeks ago—the latter part of the summer or the early fall. The samples had remained in a refrigerator, but a great many degrees above freezing, and it occurred to me that the low albumin might have been caused by the action of some enzym, for we know that whereas some of these preservatives will prevent the action of bacteria and germs, they do not in anywhere near the same degree prevent the action of the enzyms. I merely suggest this as a possible explanation.

Mr. Wheeler then presented the report of the committee on amendment of the constitution, as follows:

REPORT OF COMMITTEE ON AMENDMENT OF THE CONSTITUTION.

The committee reports favorably upon the amendment proposed to section 4. That section now reads:

There shall be appointed by the president at the regular annual meeting a referee and associate referee for each of the subjects to be considered by the association.

As amended the section will read:

There shall be appointed by the executive committee at the regular annual meeting a referee and associate referee for each of the subjects to be considered by the association.

Mr. Ewell moved to amend this section further by changing that portion after the word "referee" so as to make it read, "and such associate referees for each of the subjects to be considered by the association, as that committee may deem appropriate."

The latter amendment was accepted, and the original amendment, thus changed, was adopted, making the section read as follows:

There shall be appointed by the executive committee at the regular annual meeting a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

The committee reports favorably upon the amendment proposed to section 7, which section reads:

No changes shall be made in the methods of fertilizer analyses, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of fertilizer work to test the proposed changes.

I would say that this section was drawn up at a time when we devoted ourselves almost exclusively to fertilizer analyses; but since that time we have added analyses of cattle foods and analyses of human foods, and the amendment was proposed in order to embrace those two subjects as well as the subject of commercial fertilizers. The section will read after amendment:

No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

The amendment was adopted.

The committee also reports favorably upon the amendment proposed to section 2, to strike out the part reading—

Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution,

and to insert in place of it the following:

All persons eligible to membership shall become members ex officiis and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials.

The PRESIDENT. I suppose that if the constitution had been strictly interpreted not a single one of us would really be a member. I never signed the constitution in my life, and I do not know of anyone else who has.

Mr. EWELL. That is an amendment that will save us considerable trouble in the Department of Agriculture, because we get so many inquiries. In our replies we have always interpreted the constitution as though the words of this amendment were actually a part of it.

The amendment was adopted.

Mr. Wheeler then presented the report of the committee on resolutions.

REPORT OF COMMITTEE ON RESOLUTIONS.

The adoption of the following resolution which was offered yesterday is recommended:

Resolved, That the Association of Official Agricultural Chemists welcomes the action of the National Association of State Dairy and Food Commissioners in appointing a committee of its members to represent to our association its views upon the subject of food standards—the formulation of which has already been undertaken by this association for the guidance of its members—and upon the methods of detection of food adulteration.

Adopted.

The following resolution was presented this morning:

Resolved, That it is the sense of this association, and it strongly urges upon referees, that no method or modification of a method be submitted to general test by the association until such method or modification has been first tested by the referee himself with favorable results.

The committee recommends the following substitute:

Resolved, That it is the sense of this association that, until a proposed new method or modification of an existing method has been tested by the referee with favorable results, it shall not be submitted by him for general test by the association.

It is thought that this form would be as mandatory, that the referee would be expected to do what the association had expressed as its sense in the matter, without urging it upon him.

The substitute was adopted.

At 12.30 the convention adjourned until 2 p. m.

FRIDAY—AFTERNOON SESSION.

President Van Slyke called the meeting to order at 2 o'clock.

The PRESIDENT. We will continue the paper that was about to be presented when we adjourned—Mr. Patrick's paper on butter.

Mr. PATRICK. In the paper that I have to present as part of the report of the referee I have introduced a number of results from samples that were not sent out by the referee—samples that were used in the course of our somewhat extended study of renovated butters in the Bureau of Chemistry.

REPORT ON THE IDENTIFICATION OF RENOVATED BUTTER.

By G. E. PATRICK.

I. THE BOILING TEST.

This test I have made usually with aid of a test tube instead of a spoon, sometimes resorting to the latter, however, for corroboration or for a more distinct result as to "sputtering" in doubtful cases. I put into a 6-inch tube enough of the butter to make a depth of 1¼ to 1½ inches when melted, and boil it intermittently over a free flame, boiling it briskly for six or eight seconds, then removing it from the flame for an equal or greater length of time—long enough to be certain of its behavior—and repeating the operation until all the water is expelled. If the butter be genuine the time arrives when, after removal from the flame, it boils up in small bubbles, producing a foam which rises to a greater or less height, sometimes even overflowing the tube. Renovated butter produces much less foam, if any (oleomargarine none) and of course boils more noisily than does genuine butter.

From late experience I am inclined to believe that in doubtful cases the spoon form of the test is more reliable than the test-tube form.

This test—i. e., the boiling test in some form—being well known and in general use, I will report results only on the referee's samples A and B, and four others that accompany them throughout all the tests.

TABLE I.—*Results of boiling test.*

Sample A (No. 22608); boiled noisily, producing but little foam.
Sample 23142, process; boiled noisily, producing but little foam.
Sample 23144, process; boiled noisily, producing but little foam.
Sample B (No. 22609); boiled more quietly, producing abundance of foam.
Sample 23143, creamery; boiled more quietly, producing abundance of foam.
Sample 23145, creamery; boiled more quietly, producing abundance of foam.

II. THE HESS AND DOOLITTLE METHOD—APPEARANCE OF THE CURD AFTER DRYING ON A GLASS PLATE (MICROSCOPIC SLIDE).[a]

My first trials of this test (April, 1901) were an entire failure, because, having simply followed the authors' directions and poured the fat-free curd and brine in a thin layer upon the glass plates, the salt of the dried mass practically hid everything else from view.

In subsequent trials more satisfactory mounts were obtained by judiciously diluting the curd before spreading upon the slides; but since the brines differ greatly in their content of salt, it is impossible to know before trying how much dilution is necessary in each case. Very great dilution is evidently to be avoided, as the casein particles that lodge upon the slide are then too few in number.

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[a] Jour. Am. Chem. Soc., Mar., 1900, p. 150.

This mode of procedure, viz, diluting the curd according to judgment, always with at least several times its volume of water, before pouring upon the slides, was followed in all of the tests here recorded and in nearly all that we have made. But even with considerable experience in preparing the mounts, we frequently find them hardly satisfactory after drying, either from too much salt or because the layer of curd is too thin.

In one series of over forty samples, where we had made the layers too thick, I tried to improve them by dissolving off the salt by gentle immersion in water for a minute or so, but the result was not a success; too much besides the salt was removed or else the curd was left too transparent.

In another set, however, of four genuine and four process butters, another and more successful variation of the method was tried, viz, that of diluting the fat-free curd with considerable water, allowing to settle, and then spreading upon the slide a small portion drawn from the bottom by means of a glass tube or inverted pipette. The results on these few samples were so satisfactory that we shall try this mode again at the first opportunity. Theoretically, we should be able to escape all trouble from salt crystals in this way.

Working by the present method, however, one has often much trouble in interpreting the appearances presented by the curd. The majority of process butters, in our experience, give quite distinctly the appearance described by Hess and Doolittle, viz, a coarse or distinctly granular curd; but some samples certainly do not present this appearance distinctly. And, on the other hand, we have met with a few low-grade butters, "country butters" and "farmers' rolls," undoubtedly genuine, showing casein granules of such size and number, intermingled with fine matter of course, that if judged by this method alone they would be strongly suspected of being process butter, at least in part. In fact some samples of this class have appeared more like process butter than some few other samples known to be process butters, as shown in the following table:

TABLE II.—*Identification of process butters by the Hess and Doolittle method.*

Laboratory No.	Interpretation of appearances with—		Character of butter, as known from sources, verified by other tests.
	Power 85 diameters.	Power 7 diameters.	
22537	Suspicion of process	Suspicion of process	Country butter; packing stock.
22538	Genuine	Genuine	Do.
22539	do	Probably genuine	Do.
22540	do	Genuine	Do.
22541	do	do	Do.
22542	do	do	Do.
22549	do	do	Do.
22550	do	do	Do.
22551	do	do	Do.
22552	do	do	Do.
22553	Suspicion of process	Probably genuine	Do.
22554	do	Suspicion of process	Do.
22610	Probably genuine	Genuine	Do.
22342	Process	Suspicion of process	"Farmer's Roll," low grade.
22343	Genuine	Genuine	Do.
22345	Suspicion of process	do	Do.
22346	Probably genuine	do	Do.
22545	do	do	"Dairy Print," good quality.
22546	Genuine	do	Do

TABLE II.—*Identification of process butters by the Hess and Doolittle method*—Continued.

Laboratory No.	Interpretation of appearances with—		Character of butter, as known from sources, verified by other tests.
	Power 85 diameters.	Power 7 diameters.	
22547	Suspicion of process	Genuine	"Dairy Print," good quality.
22563	Process	do	Do.
22349	Suspicion of process	do	Do.
22543	Genuine	do	"Dairy Tub," good quality.
22544	do	do	Do.
22559	do	do	Creamery, good quality.
22564	do	do	Do.
22561	Suspicion of process	Probably genuine	Do.
22210	Genuine	Genuine	Ladled.
22214	Process	Suspicion of process	"Ladled;" appears to be in part process.[a]
22217	Suspicion of process	Genuine	Ladled.
22218	Genuine	do	Do.
22333	do	do	Do.
22335	do	do	Do.
22336	do	do	Do.
Ottumwa.	do	do	Do.
22555	Process	Suspicion of process	"Process" or renovated.
22558	do	Probably process	Do.
22560	Suspicion of process	Probably genuine	Do.
22208	do	Suspicion of process	Do.
22209	do	Genuine	Do.
21938	Process	Process	Do.
21816	Probably process	do	Do.
21817	Process	do	Do.
21794	do	do	Do.
21937	do	do	Do.
21944	do	do	Do.
22562	do	do	Do.
A (22608)	do	do	Do.
B (22609)	Probably genuine	Genuine	"Dairy."
23142	Process	Probably process	Process or renovated.
23144	Suspicion of process	Probably genuine	Do.
23143	Genuine	Genuine	Creamery.
23145	do	do	Do.

[a] Microscopic appearance strongly indicates admixture of "process."

Three different magnifying powers were used in examining the slides—a reading glass of low power, a microscopic combination enlarging 7 diameters (Hess and Doolittle employed 3 to 6 diameters), and another enlarging 85 diameters. The first was quite insufficient; the second and third each had their advantages, but a comparison of the known character of the butters with the conclusions drawn from examination with the two powers independently, and usually without knowledge of the character of the sample, shows the lower power to be in general the more suitable of the two. The higher power (85 diameters) sometimes misled by giving an exaggerated idea of the size of minute particles of casein, while on the other hand with a few samples of very smooth "process" it gave the more reliable indications. But, as the table shows, with the use of either power there was often a considerable degree of uncertainty in the conclusions, which I can hardly believe due to lack of care in making the observations or in preparing the slides. With the lower power (7 diameters) the conclusions are seen to have been positively or "probably" wrong only three times out of the fifty-three (each time on process butter); "suspicious" of what was not the fact three times; and "suspicious" (instead of being positive) of what was the fact three times. In twelve instances the appearances were so indecisive that only qualified verdicts could be rendered, and it is altogether probable that in some of these, at least, another observer would have reached other conclusions. To test this point (the personal equation of different observers) Mr. Van Dyke and Mr. Stuart made independent observations, with magnifying power of 85 diameters, on 40 samples (2 slides each), with the result that their conclusions, although expressed in some 12 cases as probabilities or suspicions, were to all intents the same in thirty-one out of the forty; somewhat different in four and exactly opposite in five instances. Their results would probably have been more concordant had the lower power of microscope been used; but the fact that different observers are likely to sometimes interpret differently the appearances upon the same slides is illustrated.

From all of our experience I conclude that while there is room for much improvement in this test, even as it stands, it is to be classed among the useful corroboratory tests available to the chemist.

III.—THE HESS AND DOOLITTLE METHOD FOR THE IDENTIFICATION OF THE SOURCE OF THE CURD BY QUALITATIVE TEST FOR ALBUMINS IN SOLUTION.

We have usually begun by at once dissolving the fat in ether without the preliminary melting and decanting of the bulk of the fat, as described by Hess and Doolittle, a variation that can not possibly have affected the tests upon the washed curd and brine, and which was adopted in order to bring the test into line with the *quantitative* curd test, and also to observe under identical conditions the behavior of the curds in regard to settling from the ether, or, more accurately, to roughly compare the amounts of that part of the curd which has the property of remaining long suspended in ether. Hess and Doolittle state that the curd of genuine butter remains suspended in ether for a considerable time.

In our experiments we have not attempted to wait for a perfect subsidence, but in each set of experiments have allowed to all the samples the same time (never more than one and one-half hours, and usually only 5 to 15 minutes) before decanting off the more or less turbid ether and adding a new portion. Proceeding in this way we found that, as a rule, at the second, third, or fourth treatment with ether, depending upon the amounts used, the remaining curds of process butters would settle immediately after stirring, leaving the ether perfectly clear, while with genuine butters treated in exactly the same way the ether would be cloudy and remain so for a long time. The treatment with ether being repeated until the sixth time, in one set of experiments, the curds of some of the genuine butters showed signs of being depleted of this particular form of matter, the ether becoming quickly clear or nearly so.

But it must be stated that while in our experience process butters nearly always behave in the manner described, we have seen several samples of genuine butter behave in exactly the same way. Therefore, while these observations are of no immediate value they may point out a line for future study of a more critical character.[a]

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[a] Further experiments made since the above was written show the phenomenon in question to be dependent merely, or at least mainly, upon the relative dryness of the curds. As might be expected, a wet curd yields less matter suspensible in ether than does the same curd when dry; and the behavior of any butter, genuine or renovated, can be entirely changed, either by air, drying it for several days in a thin layer, or by intimately mixing 10 to 15 per cent of water with the softened or melted butter.

Coming now to the test of the fat-free curd and brine, these having been diluted with a little water and the soluble portion filtered off, the clear filtrate was treated as Hess and Doolittle direct, viz, by acidifying slightly and boiling. It soon became evident, however, that the greater part of the precipitate obtained from process butters was thrown down by simple acidification, without heat. Therefore, for the sake of investigation, it has been my habit to first remove this precipitate, if any were found, and then to test the filtrate for albumin by heat. In this way I have found the albumin to be only a very minute quantity in either genuine or process butter, not nearly enough to serve as the basis for a distinguishing test. In fact, in a few samples of very rancid process butter albumin has been practically absent.

From all this it seems quite evident that the real basis of the Hess and Doolittle test is the soluble (uncoagulated) casein rather than the albumin of the brine; this, however, is nothing against the test.

Passing on to the results obtained with it, without attempting to give in detail all of the individual trials, we can say from our experience, which has been mainly with stale and rancid samples, that process butters of that description are very likely to be passed as genuine by this test, their soluble casein being often no greater in amount than that of genuine butters, and sometimes being practically absent. This may be illustrated with a set of four process and four genuine butters tested last April. The former had been kept in the refrigerator nine to twelve weeks after purchasing. Three of the latter were considerably older.

In these trials the soluble casein and the albumin were precipitated together exactly as directed by Hess and Doolittle. The amounts taken were 100 grams each.

TABLE III.—*Qualitative test of brines; soluble casein and albumin together.*

Genuine:
- 21251. Distinct milky precipitate; slow to settle.
- 21288. Mere trace; practically nil.
- 21289. Small distinct precipitate which settles.
- 21800. Very slight precipitate; less than in 21289.

Process:
- 21794. Very slight precipitate; less than in 21251 or 21289.
- 21795. Quite a precipitate; probably twenty times that from the genuine butter.
- 21802. Heavy precipitate; perhaps twice that of 21795.
- 21803. Merest trace; the least of any except 21288.

Two of the process butters would have been so declared by the test and two would not.

In Table IV is given the result with much older samples, showing the practical absence of both soluble casein and albumin in certain old process butters, and illustrating another fact not previously mentioned, viz, that some genuine (?) butters contain enough soluble casein to cause them to be either condemned by the test or regarded as very suspicious. All such samples that we have found are included in the table. With one exception they are "country butter," the "packing stock" of ladlers or renovators.

Theoretically they were probably churned from cream not very much ripened, were certainly not well freed from buttermilk at churning and were salted sufficiently to prevent, largely, the coagulation of the casein with the lapse of time. That they are exceptional among genuine butters is evident from the fact that they are the only ones giving so strong a test for soluble casein in a list of 40 genuine butters tested at one time. The other 33 (not included in the table) showed only sufficient soluble casein to be described by the words "opalescence," "faintest opalescence," "faint turbidity," and in a few cases—six—" small or light precipitate." The albumin precipitates differed somewhat among themselves, but all were very small. The samples had been in refrigerator nearly all the time since their purchase; the length of such storage is stated in the table.

TABLE IV.—*Tests for soluble casein and albumin with old butters* (*Hess and Doolittle method*).

No.	Character.	Age since purchase.		Test for soluble casein.	Test for (soluble) albumin.
		Mos.	*Days.*		
21794	Process	9	7	Considerable precipitate settled.	Slight precipitate (or cloudiness).
21816	.....do	8	4	Nothing	Do.
21817	.....do	8	4	Heavy precipitate	Do.
21938	.....do	8	3	Light precipitate settled	Do.
22208	.....do	6	8	Very light precipitate settled.	Do.
22209	.....do	6	7	Faint opalescence	Do.
22555	.....do	4	9	Nothing	Slight precipitate settled.
22556	.....do	4	9	.....do	Nothing.[a]
22558	.....do	4	9	.....do	Do.
22560	.....do	4	9	.....do	Do.
22538	Country butter[b]	4	12	Heavy flocculent precipitate.	Slight precipitate (or cloudiness).
22539	Country butter	4	12	Very heavy flocculent precipitate.	Do.
22540	.....do	4	12	.....do	Do.
22549	.....do	4	9	Heavy precipitate	Slight precipitate.
22551	.....do	4	9	Considerable precipitate settled.	Do.
22554	.....do	4	9	Heavy precipitate	Do.
22559	Creamery	4	9	Considerable precipitate settled.	Do.

[a] The tests recorded in this table were upon only 16 to 17 grams of butter. In larger amounts perhaps albumin might have been found. Casein was precipitated by 0.3 to 0.5 cc. of 10 per cent acetic acid; the filtrate from casein was merely heated for albumin.

[b] Or packing stock.

Results upon the referee's samples A and B, and the four that were tested with them throughout, were as follows:

TABLE V.—*Tests on samples of referee and comparative samples for soluble casein and albumin.*

FIRST TEST, SEPTEMBER 25.

Sample.[a]	Test for soluble casein.	Test for albumin.
A (22608). Process	Nothing	Very slight precipitate.
B (22609). Dairy	Mere trace	Opalescence.
23142. Process	Heavy precipitate	Very slight precipitate.
23144. Process	.....do	Opalescence.
23143. Creamery	Trace	Very slight precipitate.
23145. Creamery	Opalescence	Do.

[a] Samples A and B were received July 5, and had been held in refrigerator. The other four were entirely fresh, purchased September 24.

TABLE V.—*Tests on samples of referee and comparative samples for soluble casein and albumin*—Continued.

SECOND TEST, NOVEMBER 7.

Samples.	Test for soluble casein.	Test for albumin.
A (22608). Process	Nothing	Very small precipitates or turbidities for all.
B (22609). Dairy	Opalescence	
23142. Process	Small precipitate	
23144. Process	Opalescence	
23143. Creamery	do	
23145. Creamery	do	

Here it is seen that in the six weeks elapsing between the first and second tests practically all the soluble casein of 23144 and a large part of that of 23142 had disappeared, having become coagulated, no doubt.

In the process butter A this coagulation had entirely taken place before the date of the first test. Samples A and B were in refrigerator during the six weeks between the tests, the other four were in a dark closet in the laboratory. Both tests were of course made on the same quantities of butter—in this case 25 grams.

It should be stated here that a process butter made in the laboratory by incorporating clear butter fat with buttermilk two days from the churn gave a negative test for soluble casein, as would be expected. Therefore, if sour buttermilk or curdled milk of any kind be used to the exclusion of sweet milk in the making of process butter, even the fresh article would not be detected by this test.

I am informed that buttermilk is used by some of the "process" manufacturers, but whether it ever wholly replaces sweet milk I have not learned, nor am I informed as to the extent to which the "ripening" of the milk is carried before its incorporation with the butter fat; this would of course affect the results.[a]

Oleomargarine, it is known, is sometimes made with buttermilk alone, and a fresh sample of such oleomargarine submitted to this test gave the ideal result for genuine butter. The test would of course have been the same had it been a process butter made in a similar way.

IV. QUANTITATIVE ANALYSIS OF THE CURD AND BRINE (HESS AND DOLITTLE METHOD).

After what has been said concerning the qualitative testing of the curd and brine, it is only necessary here to give the results of the quantitative analyses made.

All the determinations of nitrogen in the various precipitates and residues were made by Mr. T. C. Trescot, of the Bureau of Chemistry.

In our first set of trials the soluble casein and albumin were thrown down together, exactly as directed by Hess and Doolittle. The use of the separatory funnel, as directed by them, was found not only needless but objectionable, because of adhesion of casein thereto, and was not used in subsequent trials. Fifty grams of[b] each butter were taken.

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[a] More extended experience, since the above was written, with the process butters as found in the Washington market, shows that a large proportion of these butters, even while strictly fresh, have their casein largely or wholly in the coagulated state (insoluble casein); and since most of them are doubtless made with skimmed or whole milk, and not with buttermilk, the writer infers that the ripening of the milk (for flavor) is often carried so far that the casein curdles very soon thereafter—quite likely when the milk is mixed with the hot or warm butter-fat.

[b] In later work only 25 grams were used. Even with this amount the filtration of curd is often very slow.

Results were as follows, together with the ratios regarded by Hess and Doolittle as the ratios of "casein to albumin:"

TABLE VI.—*Determination of nitrogen* (*Hess and Doolittle method*).

Sample.	Nitrogen of insoluble casein.	Nitrogen of soluble casein and albumin.	Ratio of insoluble casein to soluble casein and albumin.	Age of butter from time of purchase.	
	Per cent.	*Per cent.*		*Mo.*	*dys.*
21796. Process	0.102	0.009	11.4 : 1	2	25
21797. Process	.102	.003	36.5 : 1	2	25
21804. Process	.098	.006	15.8 : 1	2	11
21806. Process	.141	.002	69.9 : 1	2	8
21816. Process	.046	.006	7.5 : 1	1	22
21805. Oleomargarine	.082	.003	29.3 : 1	2	8
21807. Creamery	.049	.001	43.7 : 1	2	8

The ratio of 8.6 : 1 mentioned by Hess and Doolittle is approached by only two of the process butters, while two others furnished ratios averaging higher than that of the known creamery butter.

Later, in examining another set of samples, we separated the soluble casein from the albumin, precipitating the former by acetic acid in the cold, and the latter, after neutralizing the liquid, with 0.3 cc of 10 per cent acetic acid and heat.

In the filtrates from albumen the proteids and other matters precipitable by tannin were thrown down by that agent in the usual manner, and the amounts were considerable, as shown in Table VII.

TABLE VII.—*Determination of nitrogen* (*Hess and Doolittle method*).

Sample.	Percentage of nitrogen in form of—				Ratio of insoluble casein to soluble casein and albumin.	Ratio of insoluble casein to soluble casein, albumin, and tannin precipitate (protein of).	Ratio of total casein to albumin and tannin precipitate (protein of).
	Insoluble casein.	Soluble casein (by acetic acid, cold).	Albumin (by acetic acid and heat).	Tannic acid precipitate.			
22555. Process	0.011	0.039	Mere trace	0.037	0.28 : 1	0.14 : 1	1.35 : 1
22556. Process	.012	.012	.....do....	.004	1.0 : 1	.75 : 1	6.0 : 1
22558. Process	.022	.037	None.....	.029	.60 : 1	.33 : 1	2.0 : 1
22560. Process	.021	.010	Mere trace	.005	2.1 : 1	1.5 : 1	6.2 : 1
22548. Oleomargarine	.031	.038	None.....	.010	.82 : 1	.65 : 1	6.9 : 1
22566. Oleomargarine	.095	.008	Mere trace	.005	11.9 : 1	7.3 : 1	20.6 : 1
22544. Dairy tub	.066	.000	0.008	.008	8.25 : 1	4.0 : 1	4.0 : 1
22545. Dairy print	.025	.049	Mere trace	.025	.51 : 1	.33 : 1	3.0 : 1
22546. Dairy print	.031	.046	.....do....	.034	.67 : 1	.39 : 1	2.3 : 1
22563. Dairy print	.031	.017	.....do....	.024	1.8 : 1	.75 : 1	2.0 : 1
22559. Creamery	.021	.029	.....do....	.012	.72 : 1	.51 : 1	4.2 : 1
22564. Creamery	.008	.011	0.013	.024	.33 : 1	.17 : 1	.51 : 1

All of these samples had been purchased approximately two and one-half months prior to the trial, and had meantime been stored in the refrigerator.

In the first column of ratios are those called for by the Hess and Doolittle method, and which according to these investigators should be about 8.6 : 1 for process and much greater for genuine butters. A study of the table shows that neither this nor any other ratio can be found, in either of the three columns of ratios, distinguishing the one class of butters from the other.

Next we tried the method upon the absolutely fresh butters referred to above under the qualitative test, two genuine and two process, together with the referee's samples A and B, which had been held in the refrigerator since their arrival two months and twenty-one days before the trial was made. Duplicate analyses of the curd were made, the results being shown in Table VIII.

TABLE VIII.—*Determination of nitrogen in samples of referee and comparative samples (Hess and Doolittle method).*

Sample.	Percentage of nitrogen in form of—				Ratio of insoluble casein to soluble casein and albumin.	Ratio of insoluble casein to soluble casein, albumin, and tannin ppt. (proteid of.)	Ratio of total casein to albumin and tannin ppt. (proteid of).
	Insoluble casein.	Soluble casein (by alum).	Albumin (by acetic acid and heat).	Tannic-acid precipitate.			
A (22608). Process (1)....	0.073	0.005		0.008	14.6 : 1	5.6 : 1	....
(2)....	.075	0.011	0.000	.004	6.8 : 1	5.0 : 1	21.5 : 1
B (22609). Dairy (1)....	.033	.010		.010	3.3 : 1	1.6 : 1	....
(2)....	.031	.018	.000	.006	1.6 : 1	1.3 : 1	8.1 : 1
23142. Process (1)....	.009	.050		Lost.[a]	.18 : 1	.16 : 1	....
(2)....	.008	.055	.002	Lost.[a]	.10 : 1	.12 : 1	6.3 : 1?
23144. Process (1)....	.010	.065		.010	.15 : 1	.13 : 1	....
(2)....	.009	.076	.002	.006	.12 : 1	.11 : 1	10.6 : 1
23143. Genuine (1)....	.018	.021		.011	.86 : 1	.56 : 1	....
(2)....	.020	.029	.007	.006	.56 : 1	.48 : 1	3.8 : 1
23145. Genuine (1)....	.040	.011		.009	3.6 : 1	2.0 : 1	....
(2)....	.040	.021	.003	.006	1.7 : 1	1.3 : 1	6.8 : 1

[a] Assumed as 0.008 in calculating ratios.

Here again we look in vain for any uniformity of ratios by which to distinguish the two classes of butters.

Neither is there seen any marked difference between the fresh and the stale genuine butters; but the fresh process butters show percentages and ratios quite different from those of the stale "process" of this and the preceding tables, because of the much greater proportion of soluble casein; i. e., the still uncoagulated casein of the milk used in their manufacture.

Thus the conclusions drawn from our experience with the qualitative test and stated above are fully borne out by the figures obtained in the quantitative work.

V. MICROSCOPIC EXAMINATION OF BUTTER WITH AND WITHOUT POLARIZED LIGHT AND SELENITE PLATE.[a]

While some process butters have been found showing imperfect crystallization, giving the appearance of white flakes, or in lower degree of frostiness or haziness when viewed by polarized light with crossed nicols, quite a number of samples have been encountered during the past year that were nearly or quite free from these appearances, quite as much so as rather old samples of genuine butter frequently are, especially if they have been misused in regard to temperature.

In fact, a very considerable number of process butters have been found much freer from these appearances than were certain samples of genuine butter (canned) that had traveled in the Tropics, and whose frosty and flaky appearance at once raised suspicion of adulteration with foreign fats or, at least, of "processing," both of which suspicions were fully removed by a critical study of the butters. The fact seems to be that in the present practice of the best processing factories the cooling of the melted fat is effected so quickly that even incipient crystallization is almost entirely prevented; in fact, practically speaking, is prevented. When less care is taken in this step of the process the product is, of course, more easily detected by the method in question. Fresh samples are doubtless more free from these appearances than are old ones. As to colors with the selenite plate, the purple mottling which at one time I supposed to be a sign of "process" I have ceased to regard as of any significance, since it may occur in almost any sample of butter where rancidity is developing; but, strange to say, it is once in a while nearly absent in very rancid samples.

An important feature of the microscopic examination of butter is the observation of the casein, which occurs either nearly dry or inclosed in what appear to be minuet pools or droplets of brine.

In process butters especially these casein particles are usually much larger, as well as more numerous, than in most genuine butters; but I have found them of large size and in considerable abundance in a number of "country butters," some of which are mentioned in connection with the microscopic examination of curd in the Hess and Doolittle test.

Probably any butter from which the milk is not well washed at churning will show these large casein spots. In my experience good creamery butters are very nearly free from them. These spots are almost always of a pale yellow color, sometimes of a dirty white (rarely white or colorless) when viewed with a full illumination, becoming white and gelatinous in appearance when viewed without substage illumination; that is, upon simply turning away the mirror.

In fact, when viewed in this way many small casein spots frequently become visible that were not noticeable with full illumination. This examination is best made with ordinary light, the light thus being better and the field larger than when the nicols are attached.

Magnifying powers of 85 or 120 diameters give good results. With process butters the pale yellow casein spots are usually the most striking and the most characteristic objects in the field, but several mounts should be examined, as the casein is often not evenly distributed. Considering the importance of this observation of the casein, I venture to include it in my report, although not called for by the referee.

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[a] Journal Am. Chem. Soc., June, 1900, p. 328.

TABLE IX.—*Microscopic test of butters with and without polarized light.*

[Magnification, 85 diameters.]

Sample.	With substage illumination.	No substage illumination.	Polarized light; crossed nicols.	Polarized light, with selenite plate.	Conclusions and remarks.
A. Process....	Many small and some large casein spots of dirty yellow color. B. fat rosettes.	Much casein; gelatinous.	Haziness, frostiness, flakes, imperfect and perfect fat crystals.	Fat crystals, variegated colors. Purple mottles on blue field.	The abundance and size of casein spots indicate renovated or low-grade dairy butter. The fat crystals suggest renovated butter,
B. Dairy	A few pale yellow casein spots, mostly small; normal.	A few casein spots, gelatinous; normal.	No crystals, no flakes, no frostiness; normal.	Nearly solid colors. Little purple mottling on blue field.	The casein spots might come from admixture of renovated butter; they call for further study of sample.
23142. Process.	Many large casein spots, pale yellow.	Many large casein spots, gelatinous.	No crystals, no flakes, no frostiness; normal.	Solid colors, no crystals, no mottling; normal.	The numerous and large casein spots indicate renovated or low-grade dairy butter.
23143. Creamery.	Normal.......	Very few and very small casein spots; normal.	No crystals, no flakes, no frostiness; normal.	Faint purple spots on blue field; normal.	Genuine butter.
23144. Process.	Many large casein spots, pale yellow.	Many large casein spots, gelatinous.	No crystals, no flakes, no frostiness; normal.	Slight purple mottling on blue field; normal.	The numerous and large casein spots indicate renovated or low-grade dairy butter.
23145. Creamery.	Normal.......	Practically no casein spots; normal.	No crystals, no flakes, no frostiness; normal.	Faint purple spots on blue field; normal.	Genuine butter.

VI. THE "CREEPING TEST."

The "creeping test," as I venture to call it for brevity, the writer had never tried nor heard of prior to receiving the referee's circular, and therefore has little experience to offer. Four grams of the butter were dissolved in 20 cc ether and filtered into 3-inch porcelain evaporators. The appearance of the liquid was observed after ten or fifteen minutes, and of the residual fat after the ether had evaporated, i. e., next day. The results obtained on nine genuine and seven process butters and two oleomargarines are given in Table X:

TABLE X.—*Results of "creeping test."*

Sample.	Height of creeping at end of 15 minutes.	Height of creeping after standing over night.
Process, A 22608	One-half inch	1 inch.
Dairy, B 22609	Remained level	Level.
Process, 23142	One-half inch	1 inch.
Creamery, 23143	Remained level	Level.
Process, 23144	One-half inch	1 inch.
Creamery, 23145	Remained level	Level.
	Height of creeping at end of 10 to 15 minutes.	Height of creeping after standing one hour.
Dairy, 22540	Level	Level.
Dairy, 22544	do	Do.
Dairy, 22545	do	Do.
Dairy, 22546	do	Do.
Oleomargarine, 22548	One-half inch	Three-fourths inch.
Process, 22555	do	One-half inch.
Process, 22556	do	Three-fourths inch.
Creamery, 22559	Level	Level.
Process, 22560	One-half inch	One-half to three-fourths inch.
Process, 22562	do	Three-fourths to 1 inch.
Creamery, 22564	Level	Level.
Oleomargarine	One-half inch	One-half to three-fourths inch.

These results marked so clearly the difference between the genuine and the process butters tested that we had great hopes this would prove an "absolute" test, but upon applying it to a series of canned butters from the Tropics the results obtained were so out of harmony with the indications of the boiling test that we lost confidence in it. Some unknown factor seemed to influence the results. Unfortunately lack of time prevented further study of the test. It is certainly worthy of careful investigation.

VII. THE WATERHOUSE TEST.

This test, first proposed as a test for oleomargarine, was made public at the December, 1900, meeting of the American Chemical Society by Prof. C. L. Parsons, Mr. C. H. Waterhouse, the originator of the test, having died some months before, after having communicated the test to Professor Parsons.

Through the courtesy of the last-named gentleman the writer was enabled to study the test some weeks prior to its appearance in the journal of the society,[a] and the results of this study have been published in brief in Farmers' Bulletin No. 131,

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[a] March number, 1901, p. 200.

United States Department of Agriculture. As there stated, it was found that not only does oleomargarine respond to the test, but also process butter, and even some samples of genuine butter (apparently those that have been more or less melted), unless the temperatures of the test be carefully regulated.

Butter fat solidifies so much more quickly when cooled to a temperature below its hardening point than does oleomargarine that, being kept well broken up by the stirring required in the test, it will, if cooled rapidly enough, solidify in small granules, while oleomargarine will gather in a soft lump, unless the cooling be so very rapid and extreme as to granulate even the oleomargarine. But if the cooling be less rapid (by cooling with water less cold), then, under the conditions just supposed, process butter, or butter fat separated from the curd of butter, will also gather in a soft lump instead of granulating, and some genuine butters will do the same—apparently those that have been more or less melted. It may be that there are other causes for this, such as a scant amount of curd in the butter, but we have not yet had time to study this point.

In the original Waterhouse test milk is the medium in which the melted fat floats while it is cooled and stirred. Dr. Wiley suggested the use of water, and in all of our later trials we have used that medium.

It is probable that some genuine butters of the doubtful class would not behave the same in water as in milk, because of the fat of the latter, and perhaps also because of its proteid matter.

Our mode of operating the test is as follows: 100 cc distilled water in a small tin cup, 2¾ inches in diameter, are heated to just below 50° C, a slightly rounded teaspoonful of the butter is added, and gently stirred until melted; the temperature is then raised to exactly 50°, the cup is immediately placed in a pan 9 to 9½ inches in diameter at the base, containing 1,000 cc of water at 12° C. The contents of the cup are then stirred rapidly with a wooden rod (more slender than a match) for 10 minutes with a circular and cross-wise motion in turn, in such manner as to keep the fat well agitated. The water in the pan is thoroughly mixed once every minute, the cup being used as a stirrer. At the end of 10 minutes' stirring the test is finished. A butter that is found to be granular (it may be so finely granular as to appear smooth) is above suspicion of having been "processed." Such, at least, is the result of our experience to the present time. A butter which gathers in a lump must be examined by other methods to decide whether it be "process" or not.

The Waterhouse test, applied as here described, is therefore useful as a means of proving whether or not a butter is surely genuine.[a]

In conclusion I wish to give credit to my two assistants, Mr. J. H. Van Dyke, and Mr. D. Stuart, for their faithful and efficient aid in the work here recorded.

The PRESIDENT. Those who are interested in butter certainly can not help being gratified that the Department has taken this matter up in such a thorough manner, at least pointing out and making clear some of the difficulties that have to be met.

We have reached the hour which was specially set aside for the report of the committee on nominations, which will now be in order, and will be presented by Mr. Huston.

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[a] We have recently encountered (April, 1902) two "straight" oleomargarines, made in Holland, that granulate, instead of gathering, in this test—a most surprising result, as yet unexplained.

REPORT OF COMMITTEE ON NOMINATIONS.

Your committee on nominations presents the following names:

For President, H. J. Wheeler; for vice-president, R. J. Davidson; for secretary, H. W. Wiley; for additional members of the executive committee, C. G. Hopkins, of Illinois, and F. D. Fuller, of New York.

On motion of Mr. Frear the report was received and the chairman of the committee on nominations, in the absence of the secretary, was instructed to cast the ballot of the association for the persons nominated. The chairman of the committee cast the ballot, and the persons named were declared duly elected as officers for the coming year.

The President. Next in order will be the report on tannin.

REPORT ON TANNIN.

By W. K. Alsop, *Referee.*

The referee sent samples of chestnut extract, solid quebracho extract, vienna hide powder (marked No. 1), and dry chromed hide powder (marked No. 2), to fifteen chemists, requesting that analyses be made by the official method, and also after the following directions:

QUANTITY OF MATERIAL.

Use such quantity as will give 0.35 to 0.45 gram tanning material per 100 cc. Dissolve as in official method.

NONTANNINS.

(1) Prepare 20 grams of hide powder (No. 1) by digesting three days with 500 cc water, adding on each day 0.2 gram of chrome alum in solution. Wash by squeezing through linen, continue washing until no precipitate is obtained in wash water with barium chlorid. Squeeze hide in press and proceed exactly as in official method.

(2) To 14 grams of dry chromed hide powder (No. 2) in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently. Shake on shaker 15 minutes, throw on funnel with cotton plug in stem; evaporate 50 cc.

Determinations were requested on both dilutions of the extracts with wet chromed hide, and on the weaker solution by dry chromed hide.

Mr. Geo. P. Craighill last spring called the referee's attention to a method of analysis involving the use of chromed hide powder. He is using it in his laboratory with very satisfactory results. It is based on the method proposed by Mr. B. Weiss, of Vienna. As stated in an article in the Leather Trades Review, the essential details of his method are as follows:

To every 100 grams hide in 2 liters of water are added 3 grams chrome alum in solution, this alum solution being added in equal parts on three successive days. The hide is then washed until free from sulphates. To the last wash water some formalin is added. The hide is then pressed slightly and kept in a damp chamber.

Mr. Weiss states that he has kept it in that manner as long as two months, without decomposition or other change.

The nontannin determination is carried out as follows: To 130 cc of tanning solution (0.6 to 0.8 gram solids per 100 cc) add an equivalent of 7 grams dry hide, stir frequently, let stand overnight, squeeze through muslin filter, and evaporate 100 cc.

Mr. Craighill modifies the method as follows: After chroming he keeps the hide in water to which some formalin has been added, squeezing enough hide for each determination, then he proceeds as in the official method, using shaker. He also states that he has kept hide some time in water with formalin in it without any change, but that he found hide deteriorated when kept as described by Mr. Weiss.

Mr. Craighill further states that he has prepared a dry chromed hide powder that retains the power of absorbing tannin sufficiently for use in analyses. Mr. Weiss says that he was unable to prepare such a powder. Mr. Craighill proceeds as follows:

> The washed chromed powder is lightly squeezed, then thoroughly broken up and washed with alcohol, squeezed, the washing repeated, squeezed again and carefully "woolled," so that no lumps are left, then it is washed in ether, squeezed by hand and again carefully "woolled." The drying is done at ordinary temperature. It is essential that the finished product be entirely free from hard lumps, which condition can only be secured by extreme care during the process.

The dry chromed powder sent out with the samples was furnished by Mr. Craighill. It appears to be essential that hide so prepared be digested some time in the tannin solution and then shaken vigorously. This powder is free from solubles and by its use the correction for water introduced with the wet hide is avoided and the method simplified.

The most important claims for chromed hide, either wet or dry, are, that the soluble substances in the hide are rendered insoluble, and either for that reason or some other, not explained, it causes hide powders to give concordant results that otherwise differ widely by the method now in use.

This is an established fact, and for that reason alone the referee considers that he is justified in urging that the use of chromed hide be made official. A summary of the results of the cooperative work on samples follows.

TABLE 1.—*Chestnut extract—Official method (0.8 gram per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*		*Grams.*	*Per cent.*	*Grams.*	
G. P. Craighill	53.69	46.48	44.68	1.80	15.86	28.82	No. 1, official	40.00	65.4	13.84	8 hours, 100° air bath.
					15.19	29.49	No. 1, chromed	38.00	64.4	13.53	Do.
					14.76	29.92	No. 2, dry chromed	......	......	14.00	Do.
H. T. Wilson	54.32	46.00	44.13	1.87	15.50	28.63	No. 1, official	36.00	64.68	12.70	Do.
					14.13	30.00	No. 1, chromed	44.20	71.92	12.40	Do.
					15.09	29.04	Laboratory, official	44.40	73.16	11.90	Do.
					13.38	30.75	Laboratory, chromed	42.00	68.45	13.25	Do.
H. C. Reed	54.11	45.58	44.79	.79	14.58	30.21	No. 1, official	40.40	68.60	12.68	8 hours, 105° air bath.
					14.02	30.77	No. 1, chromed	37.54	66.30	12.65	Do.
					14.07	30.72	Laboratory, chromed	34.44	65.70	11.81	Do.
F. P. Veitch	54.51	45.36	44.39	.97	15.02	28.37	No. 1, official	70.00	80.60	13.58	4 to 7 hours, 70° in vacuo.
W. H. Krug	54.70	46.34	44.83	1.51	14.44	30.39	do	......	......	......	10 hours, 70° in vacuo.
					15.61	29.22	Laboratory, official	......	......	......	Do.
					14.10	30.73	Laboratory, chromed	......	......	......	Do.
F. H. Yocum	......	46.08	45.57	.51	15.39	30.18	No. 1, official	50.00	76.4	11.80	24 hours, 96° steam oven.
					14.13	31.44	No. 1, chromed	50.00	71.7	14.15	Do.
F. H. Small	54.53	45.56	44.76	.80	14.65	30.11	No. 1, official	45.50	72.9	12.33	Do.
					14.03	30.73	No. 1, chromed	45.20	73.4	12.02	Do.
J. N. Hurty	53.19	46.77	45.70	1.07	16.77	28.93	No. 1, official	51.92	75.71	12.61	8 hours, 110°.
					15.90	29.80	No. 1, chromed	58.85	81.28	11.02	Do.
W. H. Teas	......	45.94	45.17	.77	14.00	31.17	No. 1, official	50.00	70.00	15.00	18 hours, 96° hot-water oven.
					13.30	31.87	No. 1, chromed	50.00	79.00	10.50	Do.
Kerr and Mosbaugh	......	48.04	44.42	3.62	15.71	28.71	No. 1, official	......	......	......	
M. S. McDowell	......	46.60	44.10	2.50	14.88	29.22	do	49.50	......	......	24 hours, hot-water oven.
C. H. Brown	......	45.33	44.00	1.33	14.28	29.72	do	64.00	80.40	12.54	24 hours, 96° hot-water oven.
					14.06	29.94	No. 1, chromed	55.00	77.50	12.38	Do.
					14.11	29.89	No. 2, dry chromed	......	......	14.00	Do.

W. K. Alsop		45.66	44.22	1.44	14.58	29.64	No. 1, official	64.00	80.50	12.48	Do.
					14.41	29.81	No. 1, chromed	55.00	75.80	13.31	Do.
Average		46.13	44.67	1.46	15.05	29.62	No. 1, official				
					14.35	30.32	No. 1, chromed				
Highest		48.04	45.70	3.62	16.77	31.17	No. 1, official				
					15.90	31.87	No. 1, chromed				
Lowest		45.33	44.00	.51	14.00	28.37	No. 1, official				
					13.30	29.49	No. 1, chromed				
Greatest difference		2.71	1.70	3.11	2.77	2.80	No. 1, official				
					2.60	2.38	No. 1, chromed				
Greatest difference from average.		2.38	1.03	2.16	1.72	1.55	No. 1, official				
					1.55	1.55	No. 1, chromed				

TABLE. 2.—*Chestnut extract—Experimental method (0.35 to 0.45 gram tanning material per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*		*Grams.*	*Per cent.*	*Grams.*	
G. P. Craighill	53.69	46.26	44.22	2.04	15.42	28.80	No. 1, official..........	40.00	68.4	12.64	8 hours, 100° air bath.
					14.52	29.70	No. 1, chromed	36.00	64.4	12.82	Do.
					14.50	29.72	No. 2, dry chromed ...	....	....	14.00	Do.
H. T. Wilson	54.32	45.63	44.34	1.29	15.51	28.83	No. 1, official..........	36.55	64.46	12.99	Do.
					14.40	29.94	No. 1, chromed	41.00	69.85	12.36	Do.
					13.99	30.35	No. 2, dry chromed ...	....	....	[a] 7.00	Do.
					14.08	30.26	.....do	....	....	[a] 7.00	Do.
					14.33	30.01	No. 3, laboratory, official.	48.00	72.31	13.29	Do.
					13.63	30.71	Laboratory, chromed.	40.00	68.45	12.62	Do.
H. C. Reed	54.11	45.75	44.95	.80	14.25	30.70	Laboratory, official ...	45.90	72.2	12.76	8 hours, 105° air bath.
					13.97	30.98	No. 1, chromed........	38.33	69.1	11.85	Do.
					14.00	30.95	No. 2, dry chromed ...	....	....	14.00	Do.
F. P. Veitch............	54.51	45.59	44.83	.78	15.27	29.56	No. 1, official..........	70.00	80.6	13.58	4 to 7 hours, 70° in vacuo
					15.72	29.11	No. 2, dry chromed ...	....	....	14.00	Do.
F. H. Small	54.77	45.52	44.41	1.11	14.35	30.06	No. 1, official..........	45.4	72.9	12.30	24 hours, 96° steam bath
					14.12	30.29	Laboratory, official ...	50.0	78.8	10.60	Do.
					14.01	30.40	No. 1, chromed........	44.7	73.4	11.89	Do.
					14.03	30.38	Laboratory, chromed.	45.0	75.0	11.25	Do.
					14.92	29.49	No. 2, dry chromed ...	....	....	14.00	Do.
F. H. Yocum...........	52.96	47.04	46.41	.63	15.73	30.68	No. 1, official..........	....	76.4	....	Do.
					14.52	31.89	No. 1, chromed........	....	71.7	....	Do.
M. S. McDowell	....	46.13	44.07	2.06	13.38	30.69	No. 1, official..........	51.0	....	....	24 hours, hot-water oven.
					14.37	29.70	No. 1, chromed........	50.0	....	....	Do.
W. H. Teas.............	....	46.00	44.86	1.14	14.05	30.81	No. 1, official..........	50.0	70.0	15.00	18 hours, hot-water oven.
					13.76	31.10	No. 1, chromed........	50.0	79.0	10.50	Do.

C. H. Brown	....	45.37	44.00	1.37	14.47	29.53	No. 1, official	64.0	80.40	12.54	24 hours, 96° hot-water oven.
					13.83	30.17	No. 1, chromed	55.0	77.5	12.38	Do.
					14.18	29.82	No. 2, dry chromed	....	....	14.00	Do.
W. K. Alsop	....	45.59	45.00	.59	14.46	30.54	No. 1, official	64.0	80.50	12.48	Do.
					14.01	30.99	No. 1, chromed	55.0	75.80	13.31	Do.
					14.06	30.94	No. 2, dry chromed	....	....	14.00	Do.
Average	54.06	45.88	44.70	1.17	14.74	29.96	No. 1, official	....	....	....	
					14.15	30.55	No. 1, chromed	....	....	....	
					14.43	30.27	No. 2, dry chromed	....	....	....	
Highest	....	47.04	46.41	2.06	15.73	30.81	No. 1, official	....	....	....	
					14.52	31.89	No. 1, chromed	....	....	....	
					15.72	30.95	No. 2, dry chromed	....	....	....	
Lowest	....	45.37	44.00	.59	13.38	28.80	No. 1, official	....	....	....	
					13.76	29.70	No. 1, chromed	....	....	....	
					13.99	29.11	No. 2, dry chromed	....	....	....	
Greatest difference	....	1.67	2.41	1.47	2.35	2.01	No. 1, official	....	....	....	
					.76	2.19	No. 1, chromed	....	....	....	
					1.74	1.84	No. 2, dry chromed	....	....	....	
Greatest difference from average.	....	1.16	1.71	.89	1.36	1.16	No. 1, official	....	....	....	
					.39	1.34	No. 1, chromed	....	....	....	
					1.29	1.18	No. 2, dry chromed	....	....	....	

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[a] 100 cc of solution used.

TABLE 3.—*Quebracho extract—Official method (0.8 gram solids per 100 cc.).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.	Used in filtering soluble solids.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*		*Grams.*	*Per cent.*	*Grams.*		
G. P. Craighill	13.72	86.37	77.52	8.85	9.23	68.29	No. 1, official......	40.00	65.40	13.84	8 hours, 100° air bath.	No kaolin.
					8.43	69.09	No. 1, chromed ...	38.00	64.40	13.53	.....do	
					8.38	69.14	No. 2, dry chromed	....	....	14.00	.....do	
H. T. Wilson	16.52	82.60	74.93	7.67	9.82	65.11	No. 1, official......	46.00	68.48	14.50	.....do	Barytes.
					8.94	65.99	No. 1, chromed ...	44.00	74.59	11.17	.....do	
					9.04	65.89	Laboratory, official.	45.00	66.05	15.27	.....do	
					8.90	66.03	Laboratory, chromed.	45.00	66.46	15.09	.....do	
H. C. Reed	14.97	84.75	74.95	9.80	8.26	66.69	No. 1, official......	41.13	70.20	12.30	8 hours, 105° air bath.	10 grams koalin.
					7.91	67.04	No. 1, chromed ...	39.17	67.20	12.84	.....do	
					8.47	66.48	Laboratory, chromed.	34.84	67.20	11.42		
F. P. Veitch.........	16.17	83.64	75.62	8.02	9.17	66.45	No. 1, official......	70.00	80.60	13.58	4 to 7 hours, 70° in vacuo.	
F. H. Small	16.38	83.85	73.80	10.05	8.43	65.37	.....do	47.20	72.90	12.79	24 hours, 96° in steam oven.	Kaolin.
					7.98	65.82	No. 1, chromed ...	49.50	73.40	13.17	.....do	
W. H. Krug	16.20	85.00	76.22	8.78	7.90	68.32	No. 1, official......	....	....	....	10 hours, 70° in vacuo.	
					9.51	66.71	Laboratory, official.	....	....	....	.....do	
					9.54	66.68	Laboratory, chromed.	....	....	....	.....do	
F. H. Yocum.......	14.22	85.78	75.42	10.36	9.88	65.54	No. 1, official......	50.00	76.40	11.80	24 hours, 96° steam oven.	Kaolin.
					8.87	66.55	No. 1, chromed ...	50.00	71.70	14.15	.....do	
J. N. Hurty	15.67	84.16	74.06	10.10	10.81	63.25	No. 1, official......	54.91	77.00	12.63	8 hours, 110°	
					10.83	63.23	No. 1, chromed ...	54.74	81.28	10.28	.....do	

Kerr and Mosbaugh	14.22	85.78	71.26	14.52	10.08	61.18	No. 1, official......	....	....	....		
M. S. McDowell	....	84.32	77.37	6.95	9.65	67.72	.....do	51.00	....	....	24 hours, hot-water oven.	No kaolin.
W. H. Teas.........	....	85.43	73.12	12.31	8.57	64.55	.....do	50.00	70.00	15.00	18 hours, hot-water oven.	Kaolin.
					7.83	65.29	No. 1, chromed ...	50.00	79.00	10.50	.....do	
					7.99	65.13	Laboratory, chromed.	50.00	78.50	10.75	.....do	
C. H. Brown	14.92	84.60	73.24	11.36	8.72	64.48	No. 1, official......	64.00	80.40	12.54	24 hours, 96° hot-water oven.	10 grams kaolin.
					8.27	64.97	No. 1, chromed ...	55.00	77.50	12.38	.....do	
W. K. Alsop........	14.87	84.84	74.48	10.36	9.44	65.04	No. 1, official......	64.00	80.50	12.48	.....do	10 grams kaolin.
					8.86	65.62	No. 1, chromed ...	55.00	75.80	13.31	.....do	
Average	....	84.70	74.77	9.93	9.23	65.54	No. 1, official......	....	....	....	....	
					8.66	66.13	No. 1, chromed ...	....	....	....	....	
Highest	....	86.37	77.52	14.52	10.81	68.32	No. 1, official......	....	....	....	....	
					10.83	69.09	No. 1, chromed ...	....	....	....	....	
Lowest	....	82.60	71.26	6.95	8.26	61.18	No. 1, official......	....	....	....	....	
					7.83	63.23	No. 1, chromed ...	....	....	....	....	
Greatest difference.	....	3.77	6.26	7.57	2.55	7.14	No. 1, official......	....	....	....	....	
					3.00	5.86	No. 1, chromed ...	....	....	....	....	
Greatest difference from average.	....	2.10	3.51	4.59	1.58	4.36	No. 1, official......	....	....	....	....	
					1.97	2.96	No. 1, chromed ...	....	....	....	....	

TABLE 4.—*Quebracho extract—Experimental method (0.35 to 0.45 gram tanning material per 100 cc).*

Analyst.	Moisture.	Total solids.	Soluble solids.	Reds.	Nontannins.	Tannins.	Hide powder.	Weight of wet hide used.	Water in wet hide.	Weight of dry hide.	Method of drying.	Used in filtering soluble solids.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*		*Grams.*	*Per cent.*	*Grams.*		
G. P. Craighill	13.72	86.45	77.26	9.19	9.09	68.17	No. 1, official	40.00	68.4	12.64	8 hours, 100° air bath.	No kaolin.
					8.27	68.99	No. 1, chromed	36.00	64.4	12.81	do	
					8.34	68.92	No. 2, dry chromed			14.00	do	
H. T. Wilson	16.52	82.91	76.15	6.76	10.75	65.40	No. 1, official	46.00	67.96	14.73	do	Barytes.
					8.51	67.64	No. 1, chromed	44.00	74.59	11.17	do	
					8.59	67.56	No. 2, dry chromed			[a]7.00	do	
					8.56	67.59	do			[a]7.00	do	
					9.11	67.04	Laboratory, official.	45.00	66.34	15.14	do	
					8.34	67.81	Laboratory, chromed.	45.00	66.46	15.09	do	
H. C. Reed	14.97	85.40	77.93	7.47	8.96	68.97	Laboratory, official.	47.62	73.4	12.66	8 hours, 105° air bath.	5 grams kaolin.
					8.39	69.54	No. 1, chromed	37.88	67.9	12.15	do	
					8.83	69.10	No. 2, dry chromed			14.00	do	
F. P. Veitch	16.17	83.71	77.92	5.79	9.63	68.29	No. 1, official	70.00	80.60	13.58	4 to 7 hours in vacuo, 70°.	
					11.00	66.92	No. 2, dry chromed			14.00		
F. H. Yocum	13.90	86.10	75.90	10.20	9.94	65.96	No. 1, official	50.00	76.40	11.80	24 hours steam bath, 96°.	Kaolin.
					9.01	66.89	No. 1, chromed	50.00	71.70	14.15	do	
F. H. Small	16.45	84.21	76.15	8.06	8.73	67.42	No. 1, official	46.40	72.90	12.57	do	Kaolin.
					8.31	67.84	No. 1, chromed	46.40	73.40	12.34	do	
					8.86	67.29	No. 2, dry chromed			14.00	do	
					8.56	67.59	Laboratory, official.	50.70	78.80	12.08	do	
					8.92	67.23	Laboratory, chromed.	45.00	75.00	11.25	do	

W. H. Teas		85.50	74.07	11.43	9.09	64.98	No. 1, official	50.00	70.00	15.00	18 hours hot-water oven.	Kaolin.
					8.51	65.56	No. 1, chromed	50.00	79.00	10.50	do	
		85.41	73.94	11.47	8.61	65.33	Laboratory, chromed.	50.00	78.50	10.75	do	
M. S. McDowoll		85.34			9.72		No. 1, official	50.00			24 hours hot-water oven.	
					9.00		No. 1, chromed	50.00			do	
C. H. Brown	14.92	83.88	74.77	9.11	9.23	65.54	No. 1, official	64.00	80.40	12.54	24 hours, 96° hot-water oven.	10 grams kaolin.
					8.42	66.35	No. 1, chromed	55.00	77.50	12.38	do	
					8.71	66.06	No. 2, dry chromed			14.00	do	
W. K. Alsop	14.87	85.20	75.37	9.83	9.45	65.92	No. 1, official	64.00	80.50	12.48	do	10 grams kaolin.
					8.71	66.66	No. 1, chromed	55.00	75.80	13.31	do	
					9.01	66.29	No. 2, dry chromed			14.00	do	
Average		84.76	75.95	8.81	9.51	66.46	No. 1, official					
					8.57	67.38	No. 1, chromed					
					8.99	66.96	No. 2, dry chromed					
Highest		86.45	77.93	11.47	10.75	68.29	No. 1, official					
					9.01	69.54	No. 1, chromed					
					11.00	69.10	No. 2, dry chromed					
Lowest		82.91	73.94	5.79	8.73	64.98	No. 1, official					
					8.27	65.56	No. 1, chromed					
					8.34	66.06	No. 2, dry chromed					
Greatest difference.		3.54	3.99	5.68	2.02	3.31	No. 1, official					
					.74	3.98	No. 1, chromed					
					2.66	3.04	No. 2, dry chromed					
Greatest difference from average.		1.85	2.01	3.02	1.24	1.83	No. 1, official					
					.44	2.16	No. 1, chromed					
					2.01	2.14	No. 2, dry chromed					

[a] 100 cc of solution used.

These reports indicate—

(1) That the results obtained by the use of wet chromed hide powder are more accurate than those with unchromed hide. This is especially so in the results reported as obtained by the use of the weaker solution.

Several chemists did not report results with the dry chromed powder. Two reported that they could not get satisfactory results with it. The reports received are encouraging and indicate that the subject should be thoroughly investigated.

(2) That more concordant results can be obtained by the use of the weaker solution.

(3) That the method of drying has not much influence on the tannin result, provided the determinations in each analysis are dried in the same manner.

(4) That the determination of soluble solids is the weakest point in the method, and that some method must be adopted that chemists will follow.

Mr. Wilson reports as follows regarding the use of chromed hide powder:

> Two things are well established. Powders unlike in absorptive power become alike when properly chromed. It is possible to produce a dry chromed powder ready for use. In support of the latter statement the referee has distributed a powder which yields results of astonishing concordance. The method for its preparation is very simple, and one which powder makers may easily adopt. In the opinion of the writer, the association should take official cognizance of this feature and recommend its adoption by powder producers.

Mr. Wilson reports in regard to "unlike powders being made alike," as follows:

TABLE 5.—*Quebracho extract.*—(*Solution 0.35 to 0.45 gram tannin per 100 cc.*)

VARIOUS HIDE POWDERS, CHROMED AND UNCHROMED.[a]

Hide powder.	Volume solution shaken.	Wet hide.	Moisture.	Dry hide.	Dry hide per 100 cc solution.	Nontannin.
	cc.	*Grams.*	*Per cent.*	*Grams.*	*Grams.*	*Per cent.*
I, unchromed	200	46	67.96	14.73	7.37	10.75
III, unchromed	200	45	66.54	15.14	7.57	9.11
I, chromed	200	44	74.59	11.17	5.59	8.51
III, chromed	200	45	66.46	15.09	7.55	8.34
IV, chromed	100	25	54.36	11.40	11.40	8.23
I, chromed	200	40	74.59	10.16	5.08	8.56
Do	200	50	74.59	12.70	6.35	8.57
Do	200	60	74.59	15.24	7.62	8.57
III, chromed	200	40	71.40	11.44	5.72	8.30
Do	200	50	71.40	14.30	7.15	8.33
Do	200	60	71.40	17.16	8.58	8.33
II, dry chromed	100	....	....	7.00	7.00	8.59
Do	100	....	....	7.00	7.00	8.56
Average all chromed powders	....	....	....	....	....	8.44

[a] All dryings at 100° in air, eight hours.

Mr. Wilson further reports:

> It may be mentioned that powder No. 4 was simply a piece of white hide from the tannery, dried, rasped, degreased with ether, and chromed in the usual way. It will be noticed in Table 5 that diverse powders, after chroming, not only furnish concordant results, but may be applied in various ways and various quantities without altering the results, a condition impossible with unchromed powder. The range from the average of all the chromed powders is only 0.21 per cent, or less than 2.5 per cent of the whole percentage.

Mr. Wilson calls attention to that part of the table showing results obtained by him on hides Nos. I and III, when varying quantities of hide powders were used per 200 cc of solution, and he states that "with progressively increasing hide, no decrease of nontannin occurs."

Mr. Small reports some results obtained with a poor hide powder. He says:

> These, although not obtained by the official method, may be of interest because the hide powder used is of exceptionally poor quality, it being impossible to get results of any value with it in the raw state.

TABLE 6.—*Determination of nontannins* (*Small*).

	Chestnut extract. Nontannins.		Quebracho extract. Nontannins.	
	Chromed.	Unchromed.	Chromed.	Unchromed.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Poor hide	13.68	17.38	7.89	11.84
Hide No. 1	14.01	14.35	8.31	[a]8.73

[a] Official method.

Mr. Reed reports some analyses made with his laboratory hide, and also a hide marked "B" in the table. The nontannins only are tabulated.

TABLE 7.—*Determination of nontannins* (*Reed*).

	Unchromed.		Chromed.	
	B.	Laboratory.	B.	Laboratory.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
A solid quebracho extract, 9½ grams per liter	14.50	13.24	12.45	12.21
A solid quebracho extract, 6 grams per liter	15.10	13.18	12.02	12.23
Chestnut extract, 17½ grams per liter	15.99	....	13.68	14.07
Chestnut extract, 13½ grams per liter	15.90	14.25	13.52	....
Chestnut oak bark extract	....	22.24	....	21.38

Mr. Reed states:

> I am of the opinion that if hide powder B will answer the requirements, after being chromed pretty nearly any hide will do, for B is about as poor a hide powder as I have ever handled. That the chroming of B hide makes it fit for the analyses is abundantly proved by the result. The more analyses I make with chromed hide, the better I am pleased with the method.

Mr. Craighill has sent me results obtained with various powders unchromed, wet chromed and dry chromed. In the table following, A, B, and C are three different hide powders, the determinations being made on the same solution. Nos. 1 to 5 are results obtained on different extracts.

TABLE 8.—*Determination of nontannins* (*Craighill*).

	Nontannins.		
	Unchromed.	Wet chromed.	Dry chromed.
	Per cent.	*Per cent.*	*Per cent.*
A	20.35	19.48	....
B	24.24	19.52	....
C	22.57	19.63	....
1	20.44	18.89	18.84
		18.79	18.76
		18.77	18.84
2	17.61	16.73	16.74
3	17.73	16.52	16.76
4	17.68	16.76	16.71
5	16.75	....	15.54
			15.72
			15.70

TABLE 8.—*Determination of nontannins* (*Craighill*)—Continued.

EFFECT OF CHROMING ONE DAY AND THREE DAYS.

	Nontannins.		
	Unchromed.	Wet chromed.	Dry chromed.
	Per cent.	*Per cent.*	*Per cent.*
3 days		16.61	16.56
1 day		16.94	16.86

Mr. Yocum reports as follows on a sample of Freiberg hide powder. The dry-chromed hide was digested in the tannin solution overnight.

TABLE 9.—*Determination of nontannins in Freiberg hide powder* (*Yocum*).

	Nontannins.		
	Unchromed.	Wet-chromed.	Dry-chromed.
	Per cent.	*Per cent.*	*Per cent.*
Chestnut extract		14.81	15.02
Quebracho extract	12.15	8.93	9.01

With hide No. 1, wet chromed, Mr. Yocum's results were, with quebracho extract, 8.87; chestnut extract, 14.52.

Mr. Hurty reports the following experiments with dry-chromed hide, which was prepared as follows:

Fifty grams of hide were digested three days, with 3 grams of chrome alum in solution, 1 gram being added each day, washed free from sulphates, squeezed, digested twice with 98° alcohol to remove water, then dried. In all trials the same extract was used. The tannin solution was 1 per cent, and 10 grams of hide were used with 150 cc of solution, 50 cc evaporated. Laboratory hide was used for filter and official methods. The figures are for nontannins.

TABLE 10.—*Comparative determinations of nontannins by three methods* (*Hurty*).

Samples.	Filter method.	Official method.	Dry chromed hide.
	Per cent.	*Per cent.*	*Per cent.*
First set	16.1	17.38	16.76
			16.35
			16.60
Second set[a]			15.62
			15.66
			15.72
Third set[a]			15.52
			15.66
			15.18

Samples.	Filter method.	Official method.	Dry "formal hide."
	Per cent.	*Per cent.*	*Per cent.*
Fourth set[b]			15.18
			15.10
			15.54

[a] Dry chromed hide prepared as above, except digested six days.

[b] Obtained with "formal hide."

The "formal hide" was prepared as follows: Fifty grams of the hide was washed twice with distilled water, then treated for twelve hours with 10 cc of 40 per cent formaldehyde in 1,000 cc of water, washed once, squeezed, and treated twice with alcohol.

Mr. Hurty used twice as much chrome alum as was used in the other experiments reported. It is evident from his results that prolonged chroming with this amount of chrome tends to decrease the amount of nontannins.

The referee submits the following results, obtained on six different samples of extract, in the course of routine work. Hide powder A is Vienna hide, and B is a powder that gives very unsatisfactory results with the official method when unchromed:

TABLE 11.—*Determination of nontannins* (*Alsop*).

Hide.	Unchromed.	Wet chromed.	Three grams chrome to 100 grams hide.
	Per cent.	*Per cent.*	
1. A	16.34	14.87	Chromed 3 days.
B	18.72	14.77	Do.
2. A	16.25	15.06	Chromed 24 hours.
B	....	15.06	Do.
3. A	18.26	15.85	Do.
B	....	16.10	Do.
4. A	16.76	15.79	Chromed 5 minutes on shaker.
B	19.31	15.89	Do.
5. A	16.54	15.59	Do.
B	19.28	15.84	Do.
6. A	13.53	12.45	Do.
B	15.27	12.40	Do.

These results, beyond showing that chroming brings these two hide powders into accord, are probably not of much value, as they are from different samples of extract.

Chroming on the shaker has given very satisfactory results with some hide powders, but others plump so much that they are very difficult to squeeze afterwards. The greatest objection to the method for the use of wet chromed hide, as outlined in directions sent to collaborators, is the length of time required to chrome the hide—that is, three days. The referee considers this a serious objection, as it will delay analyses unless hide can be kept satisfactorily when chromed. This method, at best, is an experiment.

Since sending out the samples experiments have been made in order to determine whether the time can not be shortened without disadvantage.

Mr. Brown's experiments along this line follow.

TABLE 12.—*Determination of nontannins with regard to time involved (Brown and Alsop).*

BROWN.

Hide powder.	Chestnut extract (0.8 gram solids per 100 cc).	Chestnut extract (0.4 gram tannins per 100 cc).	Quebracho extract (0.8 gram solids per 100 cc).	Quebracho extract (0.4 gram tannins per 100 cc).
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Vienna, unchromed	14.28	14.47	8.72	9.23
Freiberg, unchromed	16.54	16.78		10.71
No. B, unchromed	16.47	17.12		
Vienna, chromed 3 days	14.06	13.83	8.27	8.42
Freiberg, chromed 3 days	14.76	14.34	8.20	8.23
No. B, chromed 3 days	14.04	13.91		8.41
Vienna, chromed 1 day	14.01	14.02		8.44
Freiberg, chromed 1 day	14.71	14.62	8.14	8.46
No. B, chromed 1 day	14.02	13.92		8.27
Dry chromed	14.11	14.18		8.71
Average for chromed hide				8.42
Average for chromed hide, Mr. Wilson's table, No. 5				8.44

ALSOP.

Hide powder.	Chestnut extract (0.8 gram solids per 100 cc).	Chestnut extract (0.4 gram tannins per 100 cc).	Quebracho extract (0.8 gram solids per 100 cc).	Quebracho extract (0.4 gram tannins per 100 cc).
Vienna, unchromed		14.46		
B, unchromed		16.69		
Vienna, chromed 3 days		14.01		
B, chromed 3 days		13.81		
Vienna, chromed 1 day		14.05		
B, chromed 1 day		13.80		

These results show that there is no necessity for chroming hide longer than one day, at least, for the three powders tried, and if any one has worse samples for the official method than B and Freiberg they are unfortunate. One or two chemists have expressed the opinion that three days' chroming is necessary, or at least better, but have not submitted any results that support their views.

In Table 8 the results by one day and three days' chroming show about 0.3 per cent lower nontannins for the longer period. Mr. Hurty gets lower results by long chroming with larger quantities of chrome. Assuming that Mr. Craighill's dry-chromed powder chromed one day is free from solubles, as well as the hide chromed three days by Mr. Hurty, who states that he tested for soluble hide in filtrate with negative results, what warrant is there for chroming any longer, even if the nontannins may be lowered? The great advantage of the hide powder method is that it approximates the conditions in the tannery.

Vienna hide has been considered the standard by most chemists, because until recently it gave concordant results and a nontannin filtrate free from tannin and almost free from soluble hide. No other hide powders that the referee knows of fulfill these conditions as well. Some differ widely from Vienna and almost always give higher nontannins. Chroming causes these powders to give concordant results with a somewhat lower nontannin in the case of Vienna hide when 3 grams chrome per 100 grams hide are used. The referee believes that the best way to chrome is the method that will bring concordance with the least variation from the standard now in use.

Mr. Craighill says that his experiments point to 3 grams per 100 grams of hide as the best amount to use. This bears out experiments of the referee. The referee does not believe that there will be any material difference in hides chromed one day or three days with that amount of chrome alum, but if chemists use varying amounts of chrome, much of the benefit of the method will be lost.

The referee urges that the chroming of hide powder one day (twenty-four hours), with 3 grams of chrome alum per 100 grams of hide be made the official method. This method will not delay analyses to any extent. Hide started one afternoon will be ready the next day. It is generally most convenient to dissolve extracts in the afternoon and analyze the next day.

He also advocates a provisional method for dry chromed hide and recommends that it be carefully experimented with next year; also that an effort be made to interest hide-powder manufacturers to find out if it is possible to produce this material in marketable quantities of a satisfactory quality.

The referee has decided to advocate the adoption of 0.35 to 0.45 grams tannin per 100 cc as the official method for solution of tanning materials. A comparison of tables 1 to 4 seems to warrant this decision, which is scientifically correct, in theory at least. It is the method adopted by the European association and brings our methods that much nearer in accord. The majority of collaborators who expressed any opinion declared in favor of it. Prof. H. R. Procter states:

> We have found in practice that the difficulty of choosing a quantity of material, of which the tannin will fall within these limits, to be largely an imaginary one.

In regard to the volume of solution to be evaporated for the various determinations the following comments have been made:

> Mr. *Krug:* I am not favorably impressed with the use of 50 cc for evaporation. This volume of solution leaves very little limit for error, and only doubles the errors already present and so far unavoidable.
>
> Mr. *Wilson:* The method for nontannins involves a residue too small when 50 cc is used for evaporation. In the writer's opinion, the method should state a range of weight for the residue for different materials. This can be accomplished by substituting 100 cc for 50 cc in some cases. It appears to the writer that for total and soluble solids the residue as now given need not be changed.
>
> Mr. *Teas:* I do not approve of the weaker dilution, nor the use of 50 cc for evaporation, the errors being increased when so small quantities are to be weighed.
>
> Mr. *Yocum:* I am not in favor of the use of the smaller volume, the unavoidable errors being increased by its use and the shorter time required for evaporation in no wise compensating for the greater errors introduced.
>
> Mr. *Reed:* The error resulting from the evaporation of 50 cc is multiplied, whether this error occurs in pipetting or in weighing. Without doubt this is the only disadvantage. The error is proportionately larger in the case of the nontannins. To offset this we have the following advantages:
>
> (1) The time gained in the performance of the analysis.
> (2) Time saved in process of filtration.
> (3) The error from oxidation upon reduction of time in drying is lessened.
>
> In my opinion, the advantages of using 50 cc more than offset the disadvantages.
>
> Mr. *Small:* I am in favor of using 50 cc, it being my opinion that more accurate work can be done with these smaller quantities.
>
> Mr. *Craighill:* I am unqualifiedly in favor of 50 cc for evaporation, believing it to be more accurate. The longer time required for drying the larger residues has objections, to say nothing of the point of decomposition.

Mr. Brown and the referee, when making analyses of the official samples, did each in duplicate; also evaporated two 50 cc portions for each determination from the same solution. The result of their work, which involved a large number of weighings, shows that no closer duplicate weights can be obtained than when 100 cc is used. The referee agrees with Mr. Yocum that the time gained in evaporation of the smaller volume does not at all compensate for the greater errors introduced. He does not remember seeing any figures to show that errors introduced by oxidation are lessened by the smaller volume.

Mr. Reed states as an advantage the time gained in process of filtration. He gives in his report the following experiment and its results:

> Twenty-nine solutions of quebracho extract were tested, each as follows: Five grams kaolin added to portion each solution, stirred, and 150 cc passed through and

discarded. The 50 cc following (the fourth) and the two following portions (the fifth and sixth) were evaporated. The comparison was made in order to prove whether we were obtaining, as nearly as possible, the correct soluble solids by the evaporation of the fourth 50 cc according to the then official method.

Soluble solids in quebracho extracts from the evaporation of the fourth, fifth, and sixth 50 cc filtered portions of the same solutions after discarding the first 150 cc passing through.

Number of quebracho solutions used.	Number soluble solids tested.	Kaolin used, in grams.	50 cc portion evaporated.	Number instances giving highest per cent soluble solids.	Percentage of instances giving heaviest soluble solids.
29	29	5	Fourth	13	44.83
	29	5	Fifth	11	37.93
	29	5	Sixth	5	17.24

This record does not show as great uniformity as could be desired. The percentage is in favor of the fourth 50 cc, but not very markedly so. The result would have been of greater value if the time of contact had been taken in each instance.

These figures the referee considers an argument in favor of the evaporation of 100 cc, which lessens the error in the determination of soluble solids.

The method proposed by Mr. Wilson, involving the use of 100 cc for some nontannin solutions and 50 cc for the other determinations, does not seem advisable nor one likely to be followed if adopted.

Mr. Carr, in his report last year, said:

> Owing to the sensitiveness of tanning materials to the heat employed in drying, it is believed that the use of volumes yielding residues of from 0.400 to 0.500 mg is preferable to volumes giving greater residues. It is believed that if, instead of 100 cc, 50 cc shall be used for total and soluble solids, the resultant weight may be dried to constant weight in less time and at less risk of decomposition or oxidation.

The method as modified by him also involves the use of weights for nontannins as low as 40 mg.

It is interesting in this connection to note that the time for drying was not shortened.

The quantity of material advocated by the referee for solution of tanning materials involves residues somewhat heavier than the above when 100 cc is used, but in his opinion 50 cc would cause residues too small for the best results, and therefore the use of 100 cc for all evaporations is advocated.

Mr. Krug reports the following experiments on various methods of drying:

TABLE 13.—*Determination of solids with various methods of drying.*

OFFICIAL QUEBRACHO EXTRACT.

Method of drying.	Total solids.			Soluble solids.		
	A.	B.	Mean.	A.	B.	Mean.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
10 hours in vacuo, 70° C	85.05	84.95	85.00	76.32	76.11	76.22
8 hours in air bath, 100° C	84.47	84.74	84.61	74.95	75.05	74.90
8 hours in air bath, 110° C	83.26	84.21	83.74	74.74	74.74	74.74
24 hours in water bath, 98° C	85.37	85.37	85.37	74.74	74.74	74.74

OFFICIAL CHESTNUT EXTRACT.

Method of drying.	Total solids. A.	Total solids. B.	Total solids. Mean.	Soluble solids. A.	Soluble solids. B.	Soluble solids. Mean.
10 hours in vacuo, 70° C	46.17	46.51	46.34	44.69	44.97	44.83
8 hours in air bath, 100° C	47.94	48.06	48.00	47.14	47.09	47.12
8 hours in air bath, 110° C	44.80	46.17	45.49	43.77	45.14	44.46
24 hours in water bath, 98° C	46.06	46.80	46.43	44.12	44.17	44.15

On these results Mr. Krug makes the following comment:

> The comparative moisture determinations show considerable variation, and I believe this to be due to some loss by oxidation when the determinations were made by drying in air. Drying at 110° is in almost every case unsatisfactory, giving wide variations, and this temperature is no doubt too high and causes decomposition.

Last year one method for drying was changed from eight hours at 100° to 103°, in air bath, to eight hours at 100° to 110°. No reasons are assigned for the change in temperature, and the referee knows of none that justify it.

The temperature obtainable in a hot-water oven does not exceed 100°. Therefore it seems that no method of drying should exceed 100° unless it is made the only one to be used. The referee believes that the time stated for drying in the method is in excess of that required for practical purposes, especially if the weaker solution be adopted as official.

The results on total solids in the reports received from collaborators vary so much when dried under the same conditions that any comparison of the results based on different methods of drying are not of much value.

In the report on tannin presented by J. H. Yocum in 1898 Mr. Eachus contributed some valuable results on this subject.[a]

The greatest variation in per cent of tannin, in Mr. Eachus's table, 0.43 per cent, is between duplicates both dried eighteen hours. The experience of the referee is that prolonged drying causes greater variations in weights between duplicates, due, probably, to decomposition or oxidation. It also gives slightly lower weights in most cases, but does not cause any material difference in the percentage of tannin.

The following changes in the method are recommended: From eight hours at 100° to 110°, in air bath, to six hours at 100°, and from twenty-four hours to eight hours at the temperature of boiling water, which means practically a hot-water oven where the temperature is rarely above 98°. The referee has had no experience with drying extracts in vacuo, therefore can express no intelligent opinion as to the time required.

With the exception of two, the collaborators expressed no opinion on the question of filtration for soluble solids. Mr. Craighill expresses himself as opposed to the use of kaolin or any other assistant. In a report on the subject, from Mr. Reed, the following table is found:

TABLE 14.—*Influence of time of filtration and proportion of kaolin upon soluble solids* (*Reed*).

Solid quebracho extract.	Concentration.	Kaolin.	Total solids.	Soluble solids.	Reds.	Volume of filtrate.	Time of contact.
	Grams in 100 cc.	*Grams.*	*Per cent.*	*Per cent.*	*Per cent.*	*cc.*	*Minutes.*
Analysis 1	9½	10	84.75	74.95	9.80	200	290
Analysis 2	9½	5	84.75	75.43	9.32	200	215
Analysis 3	9½	2½	84.75	75.73	9.02	200	150
Analysis 4	6	10	85.01	77.37	7.64	200	175
Analysis 5	6	5	85.40	77.93	7.47	200	135
Analysis 6	6	2½	85.40	78.37	7.03	200	120
Analysis 7	6	1¼	85.40	78.70	6.70	200	110
Analysis 8	6	0	85.01	[a]78.80	6.20	200	90

[a] Shows trace cloudiness.

Mr. Reed comments on these results as follows:

> By comparing the time consumed in filtration for soluble solids in the case of the more diluted solution with that of the less diluted, it will be observed that the former

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[a] See Bul. No. 56, Div. of Chem., Dept. of Agric., p. 106.

gives a much more rapid filtration than the latter when the amounts of kaolin are the same. At the same time the more dilute solution requires a less amount of kaolin to produce the requisite clear solution. Thus we have by employing a more diluted solution been able to reduce the amount of kaolin and the time of contact of the solution with the filter paper.

As the official method now stands for soluble solids "10 grams kaolin, previously washed in a portion of the solution, may be used when a clear filtrate can not otherwise be obtained." That this does not fully cover all possible contingencies must be true from the very fact that of two extracts of the same tanning wood one may give a clear filtrate without the use of kaolin while the other may require it. If we are comparing two such extracts for their relative tannin content, and in one case use no kaolin and in the other 10 grams of kaolin, as the official method indicates, we obtain results which are certainly not justly comparable; i. e., if we employ 10 grams of kaolin to obtain a clear filtrate, where a certain amount of kaolin is absolutely necessary, we should use 10 grams of kaolin for the solution where no kaolin is required. This must be true if with the same solution we obtain such widely divergent results in soluble solids with different proportions of kaolin. As stated by the referee in last year's report, "Absorption is largely a function of the time the solution is in contact with the paper," and this being true the addition of an equal amount of kaolin to a solution not of necessity requiring it will surely tend to make the time of contact more nearly the same as that of the solution requiring it. To recapitulate, it is the reporter's opinion that there should be some method suggested so that a uniformity in time of filtration for soluble solids could be brought about, taking into consideration the varying results with different proportions of kaolin as shown.

It may be stated that by the methods now in use clear filtrates can not be obtained with some extracts with any degree of certainty or within a reasonable time without the use of some assistant. In the absence of some better method, this association should either make no determination for reds, which is manifestly unjust, or it should adopt some method involving the least error that will insure a clear filtrate in all cases, and that method be followed for all extracts. The method as adopted last year advocated the use of barytes. Almost without exception chemists who have tried this method have discarded it. The following method is proposed:

To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, decant as much of the liquid as possible, add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc through, evaporate the next 100 cc. Double-pleated S. and S. No. 590, 15 cm filter paper shall be used.

Two grams of kaolin is sufficient to clarify extracts, therefore there is no need of using 10 grams, as the larger amount retards the filtration, and in addition introduces other errors. Although by no means perfect, this method has given good results, and does not materially affect results on an extract that can be filtered clear without kaolin.

The referee has made no experiments with the optional method for soluble solids, and therefore expresses no opinion about it.

The following modifications of the official method (Bulletin 46, revised, page 79, VIII, methods) for the analysis of tanning materials are recommended:

II. Quantity of material.—First line, read "give from 0.35 to 0.45 gram of tannins per 100 cc of solution." Sixth line, read "give from 0.35 to 0.45 gram of tannins per 100 cc of solution."

III. Moisture.—(b) 1. Read: "For eight hours at the temperature of boiling water in a steam bath."

(b) 2. Read: "For six hours at 100° in an air bath."

IV. Total solids.—For 50 cc read 100 cc.

V. Soluble solids.—Read: "Double-pleated filter paper (S. and S. No. 590, 15 cm) shall be used." Proceed as follows: To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, decant as much of the liquid as possible, add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc through, evaporate the next 100 cc.

VI. Nontannins.—Read: "Prepare 20 grams of hide powder by digesting twenty-four hours with 500 cc of water, adding 0.6 gram of chrome alum in solution. This solution to be added, one-half at the beginning and the other half at least six hours before the end of the period. Wash by squeezing through linen; continue washing until the wash water does not show any precipitate with barium chlorid. Remove as much water, etc."

The remaining operations are identical with the official method, except that "100 cc" is read for "50 cc."

To VI add the following optional method:

To 14 grams of dry chromed hide powder in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently; shake on shaker fifteen minutes; throw on funnel with cotton plug in stem; let drain; tamp down the hide in funnel; return filtrate; filter until clear; evaporate 100 cc.

The PRESIDENT. Are there any papers on tannin?

Mr. KRUG. In connection with this subject I would like to address the association for a few moments in regard to the work that was done at the last meeting of the International Association of Leather Trades Chemists.

REPORT ON MEETING OF THE INTERNATIONAL ASSOCIATION OF LEATHER TRADES CHEMISTS, 1901.

By W. H. KRUG.

The International Association of Leather Trades Chemists met at Liège, Belgium, on August 28, 1901. The changes made in the method used by that association were as follows:

(1) The adoption of Freiberg hide powder, the maximum cellulose content to be 20 per cent, and the manufacturer to mark each delivery with the percentage it contains.

(2) The adoption of the A. O. A. C. method for used tanyard liquors.

Professor Procter formally proposed the adoption of the Association of Official Agricultural Chemists' method as official, but the motion was lost, the comparative results obtained by the members indicating that it was not more exact than the filter-tube method. The subject of filtration was discussed at length, and the association was forced to the conclusion that in the determination of the soluble solids a perfectly clear filtrate is absolutely necessary. The members expressed much dissatisfaction with paper No. 605, owing to the fact that it does not give uniform results and the filtration is exceedingly slow, but no change was made pending experiments which are to be reported at the next meeting. In connection with the adoption of Freiberg hide powder a subcommittee was named which will formulate methods for testing the suitability of various hide powders and for determining the moisture, ash, cellulose, and nitrogen in them. Dr. Paessler presented a series of results obtained with chromed hide powder and showed that it possesses no special advantages. This was confirmed by Professor Procter and others. The difficulties encountered in the analysis of extracts which have been decolorized by the addition of bisulphites was discussed and it was shown that the addition of a trace of hydrochloric acid to the solution gave results from 2 to 4 per cent higher than by the ordinary method. No definite conclusion was reached by the association, Professor Procter stating that the present rules permitted the addition of acid, provided this was stated in the report.

Professor Procter presented a lengthy report dealing with the differences which to-day exist between the International and Association of Official Agricultural Chemists' methods. With a considerable number of comparative experiments as a basis he arrived at the following conclusions:

(1) That rapid cooling of the diluted extract on the whole gives a somewhat higher result for soluble solids than slow cooling, and, furthermore, a smaller mean

error, so that the advantage appears to lie with the International method, which also has the merit of rapidity and convenience.

(2) That flat-bottomed dishes possess no advantage over round-bottomed dishes, the residues drying as rapidly in the latter as in the former.

(3) That the variations, which American chemists claim to be caused by the method of cooling, are more probably due to the greater oxidation which attends the longer period of drying prescribed by the Association of Official Agricultural Chemists' method.

(4) That errors may often be caused by inaccuracy of the weights used.

(5) That the determination of the soluble solids must be made on an absolutely clear filtrate to obtain concordant results, the kaolin filtration being probably the best method known for this purpose.

(6) That the compulsory adoption of an expensive quantitative paper like S. & S. No. 590 is not warranted, it being probable that any paper free from soluble matter will be fully satisfactory when kaolin is used.

(7) That the filter-tube method gives the lowest result in nontannins, the Association of Official Agricultural Chemists' method averaging from 2 to 3 per cent higher and the accuracy being somewhat greater with the former, although in both cases the mean error may be neglected.

(8) That the Association of Official Agricultural Chemists' method is less affected by differences in the hide powder, especially when the powder is faintly acid.

As previously stated, the association decided not to abandon the filter-tube method, though the Association of Official Agricultural Chemists' method was adopted for spent liquors.

The question of using a chromed hide powder has occupied the attention of American chemists for some time, and it may be of interest to quote from Professor Procter's report.

Experiments have been made on the use of chromed hide powder, as proposed recently by Weiss, though they are not so exhaustive as might be desired. Powder was chromed in several ways, using a basic solution of chrome alum, added slowly over three days, and also by drumming for half an hour * * * but none of these gave as good results as that chromed according to Weiss's original directions.

Weiss's statement was confirmed that the powder became less absorbent when strongly pressed, a lot of powder accidentally pressed to 30 per cent of water giving with chestnut extract and Weiss's method of digestion overnight a result of 10.1 of nontannins, as compared to 7.1 by the same method when the water was reduced to only 90 per cent. With the same extract the filter method showed only 6.3 per cent. It was found that shaking by the machine for 20 minutes gave somewhat lower results in nontannins than standing overnight. In each case powder equal to 7 grams of dry powder per 100 cc was employed.

It was found that while the powder with 30 per cent of moisture was quite sweet and good after three months, that with 90 per cent was putrid in a week. I am far from saying that this use of chromed hide powder is not worth further experiment, but it does not seem to have reached a completeness which would justify us in considering it as an official method, and where the shaking machine is used it is more troublesome and probably not more exact than untreated hide powder. It is also most probable that the presence of chrome and formaldehyde would alter the ratio of absorption between different tanning materials at least to some extent.

In conclusion, I desire to read a letter recently received from Professor Procter and would suggest that the method of determining the absorption—correction for the filter paper in the determination of the soluble solids described therein—be made the subject of an investigation by the referee for the ensuing year. The letter is as follows:

Leather Industries Laboratories, the Yorkshire College,
Leeds, October 25, 1901.

Dear Mr. Wiley: We are experimenting here with a method for correcting the results of filtration of tanning liquors through different papers, which in view of the early meeting of the Association of Official Agricultural Chemists I hasten to communicate to you, although we have not yet tested it at all fully. The principle of the correction is that we adopt such a method of filtration in the first instance as will give a perfectly clear filtrate with the material in question. Suppose, for instance, that to do so we employ paper 590 and 3 grams of kaolin. In order to establish the correction for the particular material and for this special mode of filtration we make a second filtration, using double paper 590 and 6 grams of kaolin. Naturally this work must be done at least in duplicate to avoid experimental errors. In the second filtration we have practically double the true absorption which takes place in the first, and consequently the second liquor will leave a smaller residue than the first one. The difference between the two, then, is the correction required, and must be added to the result with the single filtration in order to compensate for the loss with 1 paper and 3 grams of kaolin. Of course, there is the theoretical objection that the liquor on passing through the second paper is already weaker and presumably gives up somewhat less tannin to the paper, but a little numerical consideration will show that this correction of a correction is quite too small to influence the results of analysis. The method is easy of execution, the filtration through double papers taking but little longer than that through a single one, and from the work we have already done it is evident that the results if not absolutely accurate at least very much diminish the error which the paper introduces. Of course, if this should turn out on practical experience to be the case it at once unifies our methods with regard to filtration, since for any particular extract we shall use the paper we find most satisfactory and make the corresponding correction, of which in many cases one determination will serve for a whole series of analyses, the whole thing being only a question of tenths per cent.

At the meeting of the International Association in Liège the absolute necessity of filtering to perfect clearness was strongly insisted upon, and I very fully indorse this as being an essential to concordant results. Of course, it may happen that in some cases a tanning material contains small quantities of a coloring matter or some special substance which is fully absorbed by the first paper, and in this case that substance must necessarily be reckoned as insoluble matter. Whether these cases ever arise is uncertain, but some conclusion may be arrived at by experimenting with extract infusions and liquors which are optically clear without filtration. In this case the whole loss should take place in the first filtration and no loss or a much smaller one in the second. I have not, however, yet come upon any cases which support this idea.

Hoping that some of your chemists will take up the matter and confirm or condemn the method which I suggest,

I am yours, faithfully, HENRY R. PROCTER.

The PRESIDENT. Professor Atwater asked to have a statement made on the floor which might be of interest to some individuals in the association, and Mr. Woods, I believe, was asked to make the statement. Mr. Woods has the floor.

Mr. WOODS. Of course you know the character of work Professor Atwater is doing along the line of nutrition investigations. He needs one or two men to cooperate with him in connection with that work. I do not suppose the salary would be very large, possibly $1,000 or $1,200. If there are any young men here interested along that line of work—the nutrition of man—I suggest that they write to Professor Atwater.

Mr. HOPKINS. I think it would be well to have Mr. Woods make a similar announcement for several others of us here. We are looking for men.

The PRESIDENT. We might open an exchange department.

Mr. WHEELER. I would like to make a similar announcement in connection with the Rhode Island station.

The PRESIDENT. We will proceed now to receiving the reports of the committees on recommendations of referees. First, Mr. Huston will report for Committee A.

REPORT OF COMMITTEE A ON RECOMMENDATIONS.

Committee A has to report that there are very few radical recommendations submitted. The first thing that has been called to the attention of the committee is a request to have the method of making standard hydrochloric acid corrected in the methods. I think if the secretary will take a note of it he can correct it himself. The error is plain enough when attention is called to it.

NITROGEN.

The first recommendation is that the neutral permanganate method be adopted as a provisional method. Your committee suggests that we change the phraseology and say that it be printed as a provisional method.

The second recommendation is that the alkaline permanganate method be further tested by the succeeding referee. I would move the adoption of these two suggestions.

Carried.

There is a third recommendation which is not clear to the committee. It reads:

> That a caution be inserted in the method for total nitrogen concerning the proper digestion bath of sulphuric acid with certain classes of material, or that the question be investigated by the referee.

In examining the report the only thing we can find to which this recommendation seems to refer is that sometimes the digestion is stopped too early. I believe, since it is in a form which is not at all clear, it would be well not to take any action upon this third recommendation.

POTASH.

There are two recommendations under this head.

First. That the causes of low results obtained by the official method be investigated. This relates particularly to high-grade potash salt. Second. That the milk-of-lime method be submitted for investigation for the ensuing year. I move the adoption of these two recommendations.

Carried.

There is another matter in connection with potash which has been brought to our attention by Mr. Von Herff; that is, that the method for determining the water as laid down in our official methods is entirely different from that used by the German Kali Company. I do not know that any action is necessary beyond suggesting that the referee consider the matter and make some report next year. The Kali Company heat to a low red heat, which of course is very different from our procedure.

PHOSPHORIC ACID.

There is no formal recommendation in this report. Under the head of recommendations there is a discussion as to whether it might not be well to take up the question of the availability of the phosphoric acid of basic slag, and a suggestion that the method of digestion with citric acid would be a proper basis upon which to proceed. There is, however, no formal recommendation on this, so I presume the referee will be at liberty to take up such work as he wishes next year.

ASH.

(1) It is recommended that the title of this section be changed from "Methods for the analysis of ashes" to "Methods for the determination of inorganic plant constituents." The reason is that there is no method known, at least at present, by which a plant can be burned and all the sulphur, chlorin, and other constituents

left in the ash. That is to say, we are not able to prepare an ash which contains all the inorganic constituents of the plant. I move the adoption of this recommendation.

Carried.

(2) It is recommended that the method for the determination of sulphur in plants, called the nitric-acid method, as modified, be adopted as a provisional method. The committee has taken the liberty of changing phraseology a little. There has been absolutely no change in the subject-matter:

Place 5 grams of material in a 3½-inch porcelain evaporating dish, add 20 cc of nitric acid (conc.), and heat the mixture cautiously on a water bath until all danger of overflowing is passed. Partly evaporate, add 10 cc of a 5 per cent solution of potassium nitrate, evaporate the mixture to complete dryness, and ignite, at first gently and then under a blast lamp, until the residue is white. Then dissolve in hydrochloric acid, evaporate to dryness, and heat for some time in an air bath to render silica insoluble. Take up the residue in water, with the addition of a little acid, filter, and precipitate the sulphuric acid with barium sulphate, etc., in the usual way.

I would like to ask about the following phrase: "At first gently and then under a blast lamp." The committee is in doubt as to whether it is intended that the ignition be made, as in some of the steel works, by inverting a blast lamp or not.

Mr. **Fraps**. I would say make it over a blast lamp.

Mr. **Huston**. I move the adoption of this recommendation.

Mr. **Frear**. May I inquire whether that is a recommendation of a provisional method?

Mr. **Huston**. Yes; entirely provisional; not official at all.

The recommendation was adopted.

Mr. **Huston** (resuming report of Committee A):

(3) It is recommended that the determination of chlorin be omitted from the methods until a method can be adopted which will give something like the truth.

Of course this refers to the method after the section is renamed. I do not understand that there is any particular fault found with the determination of chlorin in ash. The fault is with the determination of chlorin in the plant. I therefore move the adoption of this recommendation.

Adopted.

(4) It is recommended that the determination of potash by ignition of the plant substance with sulphuric acid, as described in the determination of potash in fertilizers, be adopted as an alternative method. I move the adoption of this recommendation.

Adopted.

The entire method of analysis has been rewritten in the report on ash, and as we had no copy of our methods here with which to compare them, the committee virtually decided that the methods as rewritten by the referee on ash be transmitted to the secretary for incorporation in the report and recommended for adoption. I therefore make a motion to that effect.

Carried.

Mr. **Veitch**. Did the committee consider the use of the Eschka method for the determination of sulphur at all? It was spoken of in the meeting yesterday.

Mr. Huston. We did not consider that method; but, of course, at the time the referee's report is under consideration here it is quite in order for any member to move that the referee be instructed to consider that method next year.

Mr. Veitch. I move that the referee be instructed to consider the Eschka method for the determination of sulphur in plants.

Carried.

SOILS.

There are no formal recommendations in connection with the report on soils; that is, in regard to the matter of soil analysis. There is, however, a recommendation that a committee be appointed to take up the subject of soil sampling, and your committee on recommendations move that such a committee be appointed. I think a committee of three would be sufficient.

Carried.

Mr. Hopkins. It seems to me that if such a thing can be done it would be well for us to try to arrive at some provisional method for sampling soils at this time. A method of sampling soils is not quite the same as laying down a method of analysis. There is a large amount of soil investigation at our own station that must be done during the coming year, and it is necessary that we at least follow some method, and I would like, for my part, some expression of opinion from the members of the association, and, if possible, that we might adopt a provisional method for sampling soils. I have written the following brief description of a method that we use, and regarding which I have talked to several members of the association.

METHOD FOR TAKING SAMPLES OF SOILS FOR ANALYSIS.

By C. G. Hopkins.

All samples of soils taken for analysis should be composite and should be composed of representative samples taken from at least five different places in the field sampled, each individual sample to be a column of uniform soil extending through the stratum sampled.

One composite sample should be taken from each important and distinctly different soil stratum to a depth of 40 inches, or 1 meter, including a composite sample from the arable stratum, or plowed soil, usually about 6 inches or 15 cm. deep.

If the plow line and the subsoil line coincide, and the subsoil is a fairly uniform stratum to a depth of 40 inches, then only two composite samples need be taken, one of the arable soil and one of the subsoil. But if the subsoil line is lower than the plow line and not below 40 inches, then both strata below the arable soil should be sampled, which would make three strata to be sampled and necessitate the taking of three composite samples from the field—one from the surface or arable soil; one from the subsurface soil, that is, from the stratum between the plow line and the true subsoil line, and one from the true subsoil.

Mr. Hopkins (continuing). I may say that the latter condition obtains in most of the soils in Illinois. We have the plow line about 6 to 7 inches from the surface, and we plow to about that depth. We have a soil that is quite similar to the arable soil, a rich, black loam, that extends to 18 or 22 inches. Then we reach the subsoil line, and below that there is usually a stiff clay. If we go down 3 or 4 feet we have three distinct types of soil—certainly upon our old, worn land; that is, we have 6 or 7 inches which has perhaps been turned and turned and mixed for from forty to one hundred years; then we have, about 12 inches below that, soil which was once somewhat similar, perhaps rich, black loam, but which has never been disturbed and which may be very rich in some constituents that have been exhausted from the plowed soil; and from 20 inches to below we have usually a rich subsoil.

I would at least like an expression of opinion from those who are familiar with this subject, and see if it is not possible to adopt some provisional method; and I would move the adoption of this method as I have suggested, perhaps with some modifications or amendments.

Mr. WILLARD. We are interested in soil more or less in Kansas, and I am very glad this question has come up. I have noticed the lack of a uniform method. I am very much pleased with what Mr. Hopkins has suggested here, and it is in line with what we have done. It seems to me that from the present light that is a very good provisional method. I second the motion.

Mr. HUSTON. I think the method outlined by Mr. Hopkins has some very good points, and that it is very well adapted to the prairie, but I do not think it a good method to use in a section where there is rock. It seems to me that we had better discuss the subject in two parts. First, the sampling of surface soil, on which I think we can agree, and take up the subsoils separately. I believe a great deal in regard to subsoil will have to be left to the discretion of people in different sections of the country.

Mr. HOPKINS. I did not mean to make any fixed regulation here for sampling the subsoil. I simply stated that representative samples should be taken from each important and distinctly different stratum. It may happen that there are three. It may happen that the plow line and the subsoil line will coincide, and in that case we would only have two samples. I have found soils where four or five different samples should be taken, because there were distinctly different strata.

The motion to adopt and print the method proposed as a provisional method was carried.

Mr. HUSTON (resuming report of Committee A):

The matter of soil sampling has virtually, then, been settled. Another question which was brought up was the precipitation of the last trace of barium by the use of ammonium sulphate in determining potash. Your committee recommend that this be referred to the next referee on soils, and I make a motion to that effect.

Carried.

The attention of the committee is also called to the matter of methods for determining soluble portions of alkali soils, and, in general, any special methods necessary for handling alkali soil; and we move that this matter be transmitted to the referee, and that he be asked to report at the next meeting what special methods may be needed, if any, in addition to our present method.

Carried.

The question of mechanical analysis of soils has been called to the attention of the committee. This has been before the association before more than once. While we recognize the importance of mechanical analysis of soils, we have not heretofore considered that a chemical subject; and while it is very closely related to our work, I think that we are hardly prepared to do more at present than to recommend that the referee take this matter up and be prepared to discuss it before the next meeting of the association. We make a motion to that effect.

Carried.

The following resolution was introduced:

Resolved, That the policy of the association as to its way of investigating analytical methods for the determination of available plant food be changed.

That in place of asking the cooperation of all chemists in the comparative study of two or more methods on a furnished set of samples, each of those chemists who are willing to participate in the work be asked by the referee to take up such a line of soil study by any method he may choose upon known soils of his own State or upon other known samples, the avowed purpose of such study being the working out of a chemical method for determining the available plant food of soils.

Further, that those who engage in this work shall keep the referee informed of their lines of work, shall furnish him with a summary of their results, to be presented by him to the association at the regular meetings, and shall from time to time, as the occasion warrants, present to the association the details of their work.

In regard to this resolution your committee believe that it is inspired by a proper motive, but we do not wish to limit the referee in his method of working. The referee has the authority to do his work in any way that he thinks best, and while we think this resolution ought to be transmitted to the referee as a suggestion or an aid in his work, we do not feel justified in moving its adoption as controlling the policy of the association or as limiting the referee, although, of course, any member of the association can move it if he wishes.

Mr. Frear. I would move its reference to the referee on analysis of soils for his consideration.

Mr. Huston. This committee will second that recommendation.

Adopted.

Mr. Huston (resuming report).

INSECTICIDES.

The next is the report of Mr. Haywood, the associate referee on insecticides. He recommends that methods 1 and 2 be continued as provisional methods. These are the two methods of which he gave you the results this morning, which seem to be reasonably satisfactory, or the most satisfactory that have been used, and to warrant further investigation. We therefore move the adoption of this recommendation.

Carried.

The next recommendation is that of the other section of the committee on insecticides in particular relation to formalin and that class of material. This recommendation is to the effect that the methods be continued, with the omission of certain nonessentials in the testing work. There are some points in connection with this to which attention is called here in regard to testing potassium cyanid and materials of that nature, which the committee do not consider essential; and therefore they recommend that the referees continue to test such materials, but that they confine their work to the essentials and limit it somewhat. We move the adoption of that recommendation.

Carried.

Mr. HUSTON. There is one other matter in connection with the report of this committee. I notice that nothing seems to have been done by anyone in testing formalin in which the cyanid process was used. The process must be some five or six years old, I should think; and perhaps one reason of its omission is that when it was translated by Allen it was very badly mutilated. The process is practically unintelligible as it stands in Allen, even in the revised edition. Some two years ago I had occasion to work on quite a number of samples of formalin and compared all the methods I could get hold of. This cyanid method seemed to be promising, and when I got the original account I found it worked out into a very serviceable, quick, and accurate method of determining formalin, very much better than any other method I could get hold of. I would like to suggest to the referee that that method be included. I can give him a description of the method, or he can get it from our annual report of last year. I believe it to be a very promising method for testing formaldehyde, and I would move that the referee be requested to look into the matter of the cyanid method for testing formaldehyde.

Mr. HAYWOOD. Before going into that, may I interrupt a minute to ask if Mr. Voorhees does not in his report call attention to the fact that formalin determination really belongs more to the food referee than it does to the referee on insecticides, and therefore he nearly goes to the length of recommending that it be entirely dropped by the referee on insecticides and referred to the referee on foods? They are carrying on nearly the same line of work.

Mr. HUSTON. The food committee, I think, are more particularly concerned in qualitative tests of formalin rather than the quantitative. I think they are hardly prepared to do any quantitative work on the small amount that is usually put in food. While attention is called in the report to the matter of which you speak, nevertheless the reporter afterwards recommends that they keep on testing formalin; so there is nothing for this committee to do but to present his recommendation.

The president brought up the question of whether this session should be continued until the work of the convention was finished, or whether it should adjourn and another session be held the following day. It being found that the hall would be required for a lecture at 4.30 it was decided to follow the latter course.

Mr. Wiley extended an invitation to the members to visit the laboratories of the Bureau of Chemistry of the Department of Agriculture, and suggested that they do so when the meeting adjourned, as some of the men would be at the laboratory to receive them and the following day the building would be closed.

The president announced that a photographer was ready to take a picture of the members, and he asked that they assemble on the front steps immediately after adjournment.

Mr. Ross moved that the president-elect appoint during the sitting of the convention the committees on recommendations of referees who are to serve during the next annual convention.

The motion was referred to the committee on resolutions.

Mr. Ewell offered the following resolution and suggested that it be referred to the referee on nitrogen for the coming year to report a definite plan of subdivision:

Resolved, That the title "Determination of nitrogen" be changed to read "Determination of nitrogen and nitrogenous bodies," and that the work on nitrogen be divided between two or more referees. Further, that the nitrogen methods be taken out of fertilizer methods and proper reference inserted in their place.

At 4.15 p. m. the convention adjourned until 9.30 the next morning.

THIRD DAY.

SATURDAY—MORNING SESSION.

The meeting was called to order by the president at 10 o'clock.

The PRESIDENT: First in order will be the report on sugar, by Mr. Ewell.

REPORT ON SUGAR.

By E. E. EWELL, *Referee.*

I promise you in advance that I shall not read any long-winded report. The report on sugar this year, as last year, consists largely of an apology, and I feel that I owe an apology to the president for having made his administration in this regard lacking in success. I accepted the place with considerable hesitation, as I knew my time was very much filled up; but it was a subject ot great interest to me, and I had hopes that I might be able to do something. I was compelled, however, to put it off from day to day until it was too late to resign with justice to anybody or to inaugurate any work that would be of any value to the association.

There are several important lines of work which ought to be taken up. It is gratifying to say, however, that they are not being neglected. This is a time when research work with regard to bringing out new methods is of more importance than the trial of methods which we now know to be lacking in certain regards. The original plan was that we should take up the methods of analysis of sugar beets alone. There is a great need of an official method, because there have been numerous disputes in the districts where beets are grown and sent to the factories on test.

You will recall that when I accepted the position of referee some years ago the subject was enlarged so as to include all soluble carbohydrates, at that time of importance in our work. I am thoroughly impressed with the idea that the change was a good one, but the only difficulty that has attended it was that there were no referees for the subheadings. That was one thing I had in mind when I asked for the amendment to the constitution which authorizes the executive committee to appoint a subreferee on such subjects when it deems wise.

The reports of the methods as now in the bulletin at the time of their last extensive revision represented very well the state of our knowledge of carbohydrates—that is, from the analytical standpoint. But much has been learned since then in regard to the methods and also as to the need of improvement in the methods. There is one very important need, and one which investigators have endeavored to supply in a measure, and that is one method of manipulation for determining all kinds of reducing sugars. It is greatly to be desired that some one shall spend most of his time for a year on this work; it would make a good year's work. I wish I could do it myself, but there is no prospect that I can.

I want to say a word in regard to the optical methods. The methods were changed just after I was referee, some years ago, and through some error—I do not know just how to explain it. I recommended that the old Clerget method and the Lindet method be dropped and the method then known as the German method be substituted. This recommendation was based on the fact that all the research work previous to that time had been done with the German method. There is really no reason why that change should have been made, and there is every reason why the German method should have been retained and the present one discarded. But inasmuch as there is research work in progress now, seeking an inverting agent without action on other carbohydrates than sucrose, it does not seem wise to make any change until methods of that kind have been perfected. Mr. Tolman is actively working on such a method at this time. I shall be glad to have him speak on that subject himself, if he desires to do so.

I have written out two or three recommendations, which refer only to the division of the work. I recommend that there be a referee for the optical methods and a referee for the study of methods for the determination of reducing sugars, with the idea that if the referee can possibly arrange with some one—perhaps an instructor in a school—or with several, who can spend some time on this branch of analytical chemistry, the results may give the analyst something as a starting point. There should also be a third referee for the analytical methods of the sugar industry. I therefore recommend that the work on sugar be divided and that associate referees be appointed for three divisions of the work, as follows:

1. Optical methods.
2. Chemical methods.
3. Special analytical methods used in sugar industry.

The president referred these recommendations to the chairman of Committee B.

The President. Are there any papers, or any remarks?

Mr. Ross. I have no special paper. I have a piece of apparatus that it will only take a minute or two to exhibit. It is used in a modification of the electrolytic process of determining invert sugars, or rather of determining the copper which is thrown down in this process. Quite a number of years ago there was a process proposed by Formanek in the Zeitschrift für Rüben Zuckerind in Böhmen, vol. 14, p. 178, and this, I think, was the first proposal that had been made to utilize the electrolytic method for estimating the copper thrown down from the Fehling solution by reducing sugars. In this original process the precipitate of copper suboxid was brought on an asbestos filter and washed thoroughly, then dissolved in 1.20 sp. gr. nitric acid, I believe, and the filter washed thoroughly to remove the copper from the asbestos, which was a very difficult undertaking. This solution, which should not contain more than 4 to 5 per cent of nitric acid, was electrolyzed, a large platinum dish being used for this solution. Other modifications of this method have been employed, involving the use of sulphuric acid and subsequent evaporation of the solution in order to expel the bulk of the nitric acid. A number of years ago I presented to this association a paper, which was also published in the Journal of the American Chemical Society, I believe, giving a modification of this method in which no attempt whatever was made to dissolve the precipitate on the asbestos. A large series of experiments which I made at that time showed that if you place the asbestos filter with the precipitate in a beaker, in contact with a platinum spiral, which was used as the anode, and if a 4 per cent nitric-acid solution was poured in and a platinum cylinder suspended in that, to act as a cathode, the copper suboxid would be oxidized in contact with the anode and would go thoroughly into solution just as the deposition took place on the cathode. In fact, the solution of the copper suboxid was always effected before the completion of the deposition occurred.

Now, the apparatus which I have here carries out this same principle, but in a little different way. Here we have a funnel tube. The original funnel tube that I had made for this purpose by Herman Kobe, in Berlin, was broken. This tube, which is almost an exact duplicate, is a funnel filter tube, and in the lower portion is a platinum spiral with one end melted through the glass. An asbestos filter is placed in this, filtration being accomplished by means of a pump, as usual. After washing is completed you turn off this stopcock, pour in a 4 per cent solution of nitric acid, insert the platinum cathode, make connections with the battery or whatever current you are using, and the oxidation of the copper suboxid takes place simultaneously with the deposition of the copper on this cathode. So that after you have accomplished the washing of the precipitate, which must be resorted to any case, the only thing further that remains to be done is to pour in the nitric acid and make the battery connections. At the conclusion of the test you can either draw off the liquid at the bottom by suction or else you can raise the cathode gradually out of the liquid, at the same time directing a stream of water upon it. Of course it is washed off with water and then with alcohol and at once dried and weighed. This apparatus has given very satisfactory results in our laboratory.

I did not prepare any figures in connection with this method, because in the preceding paper, to which I referred, I have given very full results that have demonstrated that by this process a very thorough solution and deposition of the copper took place, and I only made qualitative tests to assure myself of the complete deposition of the copper. I mention it at this time, thinking possibly it might be of interest to some members of the association.

There is another method on which I did a little work and had a few results, but I have not carried it sufficiently far to justify me in presenting anything definite on the subject. I might say in passing that the principle of this method was first suggested to me by a paper by Strache and Kitt in Monatshefte für Chemie, vol. 12, p. 380, a number of years ago. The method was applied to the determination of aldehydes and ketones. This is entirely a separate and distinct modification. I have gotten some very good results, but not enough to justify me in presenting a paper at this time. The process depends upon the fact that after we have partially precipitated the copper in Fehling's solution by means of reducing sugars, upon adding phenylhydrazine to the solution we will reduce the remainder of the copper, and a definite amount of nitrogen will be given off, and this nitrogen can be measured. I do not know whether this same principle has already been applied, but if it has I would like to be informed on the subject.

The following equation gives an idea of the chemical changes involved:

$$C_6H_5—NH—NH_2+2\ CuO = Cu_2O+N_2+C_6H_6+H_2O.$$

The PRESIDENT. If there is no further discussion on the subject of sugars, the report of Committee B will be made by Mr. Winton.

REPORT OF COMMITTEE B ON RECOMMENDATIONS.

Mr. WINTON. Committee B is ready to report, first, on the methods for foods and feeding stuffs.

FOODS AND FEEDING STUFFS.

There are a great many changes suggested here. We will have to pass them very rapidly. First of all, under the head of moisture, your committee suggests that in place of the method as given we substitute the following. I do not know whether any of you have copies of Bulletin 46, or of the methods for foods and feeding stuffs as revised in 1900 and printed in Circular 7, Division of Chemistry, United States Department of Agriculture. It will be rather hard to follow these changes unless you have a copy of the methods at hand.

Under "2. Determination of moisture," for the first sentence substitute the following:

"Dry 2 grams of the substance for five hours at the temperature of boiling water in a current of dry hydrogen, in vacuo or in the air."

But as linseed meal, cotton-seed meal, and corn products contain oils subject to oxidation, we thought it would be better to insert this clause in addition: "Linseed meal, cotton-seed meal, corn products, or other substances containing oils subject to oxidation must be dried either in hydrogen or in vacuo." That is the first recommendation. The change is that the use of air be allowed, except in the case of substances containing drying oils. I move the adoption of this substitute in the method of moisture.

Adopted.

Second, under "4. Determination of Ether Extract (b) (1), Direct Method," changes are introduced to make this procedure consistent with the determination of moisture—that is to say, it is stated here to dry before determining the ether extract. We have so worded it that the method of drying will be the same used in the former case. Change the first sentence to read as follows: "Dry 2 grams of the substance as for the determination of moisture and extract with anhydrous, alcohol-free ether in a continuous-extraction apparatus for sixteen hours." There is another slight change there. We have inserted the clause, "in a continuous-extraction apparatus." It seemed to be a little ambiguous. I move the adoption of this change.

Mr. WHEELER. I would like to ask the chairman of the committee whether Blatchford's calf food and several other mixed foods on the market contain these oils that are likely to be oxidized and how anyone is to know whether such oils are present. Suppose that we had a case of mixed food. One of my assistants inadvertently dried one in the air, extracted the fat, and obtained 5 or 6 per cent. When it was dried in hydrogen in a proper manner we obtained nearly 12 per cent. I fear this is a very serious question and think we ought to hesitate before adopting it. I think it is unwise for us to take this action without further consideration. I move, therefore, that this be laid upon the table until the next meeting of the association.

Mr. WINTON. Do you move also that we reconsider the other vote? The two methods should be the same.

Mr. WHEELER. I think that would require the same motion to reconsider. I make a motion to lay this upon the table, and will follow with a motion to reconsider.

The PRESIDENT. The motion has been made and seconded that the recommendation as read be laid upon the table for one year.

Carried.

Mr. WHEELER. I make a motion that we rescind the vote whereby we voted to adopt the recommendation relating to moisture.

The PRESIDENT. The motion is made and seconded that we rescind the action whereby we adopted the recommendation relating to moisture.

Carried.

Mr. WHEELER. I would now move that this be laid upon the table for one year, and that this matter, as well as the matter of determining ether extract, be referred to the referee.

Mr. WINTON. I think myself that this is wise. I called the attention of the committee to the absolute necessity of drying linseed meal and some of the other products in hydrogen or else in vacuo, and it was conceded by the committee that wherever such substances were present it would be necessary to dry as we have done heretofore. The only point seemed to be that the majority of the members were using the air on ordinary substances. We ourselves use hydrogen. Our only purpose was to learn the wishes of the majority in this matter. As for Blatchford's calf food, that contains a large amount of linseed meal. Our intention was that if there was any doubt whatever, hydrogen or a vacuum would be used.

The motion was put and carried.

Mr. WINTON (continuing report of Committee B):

Under the head of diastase method for the determination of starch a few changes are recommended. First, in the fifth line[a] it is recommended that we strike out the words "from" and "to 40," leaving 20cc of malt extract to be used. In order to understand this I will have to go ahead a little and point out some of the other changes. Perhaps these all might be considered together, as they touch the same matter. The kernel of the changes suggested is this: According to the official method only one digestion with malt extract is made. The material is boiled with water, then it is digested for an hour with malt extract. The German chemists and some of us here in America find that we can not in some materials possibly get the starch all changed into maltose by a single digestion. We first make the paste, then treat it with the malt extract. A certain amount of starch resists the rupturing in the first place and consequently resists the action of the diastase. In order to get it all in solution we heat a second time to boiling. * * * After the bulk of the starch is taken out these remaining starch granules are more readily brought into semi-solution and afterwards converted into maltose. The changes which we suggest are those which some European analysts follow and which some of us have found are absolutely necessary. Following the methods here described, I have been in some cases unable to get within 2 or 3 per cent of the amount of starch; and as this change does not affect the method except as to add an extra precaution, I recommend that it be adopted. I will read the changes: First, strike out 40cc and use 20cc the first time. Then instead of digesting with malt after thus boiling until a microscopic examination shows the residue free from starch, it is proposed that we digest for a definite length of time, and then without examining it with the microscope boil again. The microscopic examination is mentioned later in the paragraph. I think we might vote on this first. I move the adoption of these changes.

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[a] As printed in Circular 7, Bureau of Chemistry, U. S. Dept. Agr.

Mr. Wheeler. I rise to second that motion, but would like to ask whether the motion carries with it the retention of the microscopic examination at the end?

Mr. Winton. I noticed that omission. As it now stands, that was taken out, but I think it was an oversight, and I am quite sure that the committee will approve of the retention of the microscopic examination later on.

Mr. Wheeler. If the committee would retain that feature, I would second the motion.

Mr. Winton. The committee are agreed on that point; it will be put in later in the paragraph.

The motion was put and carried.

Mr. Winton (continuing report of Committee B):

We have some suggestions as to neutralization. As stated in the methods, the acid is nearly neutralized with carbonate of soda while still hot. It is the general experience that neutralizing that solution while hot causes decomposition of some of the carbohydrates, and we therefore recommend that we cool before neutralizing, and also that instead of sodium carbonate we use a dilute solution of sodium hydrate. I think most of us do use the latter, but sodium carbonate appears to be in the method. We therefore recommend that the solution be cooled and neutralized with sodium hydrate instead of sodium carbonate, or nearly neutralized with sodium carbonate.

Mr. Wheeler. If in order, I would like to suggest that these recommendations be printed in the proceedings and that action be deferred until the next meeting of the convention. I think we came very near making a serious mistake this morning. I think there was some good reason for using the sodium carbonate. I do not feel that the members are quite in a position to act without having the methods before them. If in order, I would move that further recommendations be printed in the proceedings and that definite action be deferred until the next meeting of the association unless there is some very urgent matter.

The PRESIDENT. Is that motion intended to apply to all of the recommendations of the committee not yet recommended?

Mr. WHEELER. Unless they concern some very urgent matter.

Mr. WINTON. I think this matter in connection with starch is urgent. If you do not consider that precaution you will not get your results. And as for the others, I will say that the originators of these methods and those who have done a large amount of work have used sodium hydrate. I know that Mr. Krug strongly advises it. I have always used it myself. As for getting the association into any trouble, I do not think it will. I think it is merely in the line of improving the methods. I must say, however, that notwithstanding the vote of the committee and the opinion of those whom we have consulted, notwithstanding that their opinion was that we dry in air in most cases, I must say that personally I would like to go slowly. But in these other matters it seems to me that the case is somewhat different.

Mr. WHEELER. I will withdraw my motion.

Mr. WINTON. I think we have here those who have had a great deal of experience in these matters; and as they are not new considerations at all—they are matters that have been thoroughly investigated—I see no reason why these changes should not be made, especially as we are about to get out a new book on the analysis of fertilizers. We think these changes ought to go into the new edition.

It was then moved, seconded, and carried that the recommendations as read by chairman of committee be adopted.

Mr. WINTON (continuing report of Committee B):

The third recommendation is a very important one. I will say that not only the methods for feeding stuffs, but many of our methods, need rewriting. There are typographical errors, and there are errors that we can not lay to the printer that should be corrected, and it was the purpose of the committee to take this opportunity to change some directions here that are obviously erroneous. Now, in making up the malt extract the directions are to digest over night. Those who have tried it get a sour malt extract. The German chemists digest two or three hours, and I think those of us who have had experience do this. Some say that even half an hour is long enough, but we stick to the time-honored method. I move, therefore, that we digest two or three hours instead of overnight in preparing the malt extract.

Carried.

The next changes are under the head of the phloroglucin or glucol method. I will say that these changes have all been carefully reviewed by Mr. Krug, who, I think, has been largely responsible for these methods. What I have to present is the result of this study on his part. The first change—I do not think that this will be intelligible, because hardly any of you have this bulletin—is that the first line be made to read "A quantity of the material, chosen so that the weight of the phloroglucid obtained shall not exceed 0.300 gram, is placed in a flask."

This change was adopted.

In the seventh line the words "by means of a separatory funnel and" are to be inserted after "added." I will say this is all Greek to me, and it seems to be to you.

Mr. Bigelow. As this seems to be a technical matter, which concerns a rather limited number of the workers who have all passed upon it, I suggest that the recommendations be read and adopted as a whole, unless some one wishes to raise an objection. I make a motion to that effect.

The President. The chairman of the committee may go ahead and read these recommendations, and then an opportunity will be given for objection or statement of opinion, and at the end a motion will be in order to adopt the whole. If there is no objection to that line of proceeding, we will do that way.

Mr. Winton (continuing report):

In the seventeenth line the words "from three to" are to be stricken out; that is to say, the idea here is merely to confine the quantity used to the amount found best suited for the purpose.

In the eighteenth line the words "in a weighing bottle" are to be inserted after "weighed."

Under the head of "Galactan Method," in the twelfth line, the word "gently" is stricken out, and the words "at 80 degrees centigrade" inserted after "bath," and the words "with constant stirring" after "minutes." I will say that Mr. Krug explained all these matters in detail, and the changes seemed desirable ones. They are based on a great deal of experience.

In the seventeenth line the words "avoiding unnecessary heating, which causes decomposition," are inserted after "bath." The referee finds that there is danger of this if you are not cautious.

In the twenty-first line the words "three hours" are substituted for "a short time." These are all of the recommendations for changes in the methods.

Mr. Bigelow. I move the adoption of these recommendations.

The President. A motion has been made and seconded that the recommendations as read be adopted. Are there any remarks?

Carried.

The committee further recommends that the referee for the ensuing year on feeding stuffs undertake a study of the König method for the determination of crude fiber. This was suggested by Mr. Fraps in his paper, and the committee recommend that the referee be requested to undertake a study of this method.

Adopted.

Mr. Winton (continuing report):

This covers all the recommendations with regard to feeding stuffs.

SUGAR.

The committee recommend that the work on sugar be divided and that associate referees be appointed for three divisions of the work, as follows: First, optical methods; second, chemical methods, and third, special analytical methods used in the sugar industry. These are Mr. Ewell's suggestions.

Adopted.

I believe Mr. Krug has the recommendations in regard to tannin.

TANNIN.

Mr. Krug. I do not believe that it is necessary to read these all in detail. There are a number of minor changes in the methods, merely in the wording, to cover the recommendations of the referee. The dilution is changed from 0.8 gram per 100 cc to a constant quantity of tannin, the amount of material to be taken being varied. These are minor changes and are submitted for incorporation in the printed report.[a]

In "soluble solids," paragraph 5, the whole paragraph has been completely rewritten, as follows:

> Double pleated filter paper (S. and S., No. 590, 15 cm) shall be used. To 2 grams of kaolin add 75 cc of the tanning solution, stir, let stand fifteen minutes, and decant as much as possible. Add 75 cc more of the solution, pour on filter, keep filter full, reject the first 150 cc of filtrate, evaporate the next 100 cc and dry. Evaporation during filtration must be guarded against.

The optional method, which was a double precipitation of lead acetate, is stricken out.

In paragraph 6, on nontannins, the line beginning with the word "prepare" and ending with the word "linen" is stricken out and the following is substituted:

> Prepare 20 grams of hide powder by digesting twenty-four hours with 500 cc of water and adding 0.6 gram chrome alum in solution, this solution to be added as follows: One-half at the beginning and the other half at least six hours before the end of the digestion. Wash by squeezing through linen, continue the washing until the wash water does not give a precipitate with barium chlorid.

Throughout the method, in agreement with the recommendations of the referee, 100 cc has been substituted for 50 cc to allow for evaporation.

In the determination of nontannins the following provisional method is recommended to be added:

> To 14 grams of dry chromed hide powder in a shaker glass add 200 cc of the tanning solution; let stand two hours, stirring frequently; shake fifteen minutes; throw on funnel with a cotton plug in the stem, let drain, tamp down the hide powder in the funnel, return the filtrate until clear, and evaporate 100 cc.

These cover all the changes in the methods.

The committee further recommends that the referee for the ensuing year be instructed to investigate methods for the determination of the acidity of tan liquors. This last recommendation is in the line of expansion. The gentlemen who are especially interested in these analytical methods feel that the determination of the tannin is in very good shape at present; the method is quite exact; the work shows good results, and they would like to have the association take up a new line. The methods that are at present used in the various laboratories in the determination of such a simple matter as the total organic acids, which are formed by fermentation in the liquors used in the tannery, are of such a varied character that we think the time has come when some method should be tried and worked out, and the various methods that have been used should be compared and the best one chosen for this work.

Adopted.

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[a] These minor changes were as follows:

II. Quantity of material: First line, substitute 0.35 to 0.45 for 0.8.

Second line: Substitute "tannin" for "solid."

Sixth line: Substitute 0.35 to 0.45 for 0.8, and "tannin" for "solids."

III. Moisture. (b) 1: Substitute "eight" for "twenty-four."

(b) 2: Substitute "six" for "eight;" "100° C." for "100° to 110° C."

IV. Total solids: For 50 cc read 100 cc.

Mr. Winton. I understand there are no recommendations with regard to the methods for dairy products. Am I right?

The President. The only recommendations are for the continuation of work along similar lines.

Next in order is the report on food adulteration, by Committee C, which will be presented by Mr. Bigelow:

REPORT OF COMMITTEE C ON RECOMMENDATIONS.

FOOD ADULTERATION.

There are three recommendations. First, regarding the designation of the referee. In former years the subject was called fermented and distilled liquors, although the association never took any interest in distilled liquors. Some methods were offered in 1896, adopted provisionally, and never taken up again. Fermented liquors are never studied, with the exception of wines. Now that the subject of liquors is only one division, and perhaps a relatively unimportant division of the whole subject which the referee covers, the committee recommends that the title be changed to "referee on food adulteration." I would move that this recommendation be adopted.

Adopted.

Regarding the reports made by the referee, it was stated that they were considered by all of the referees together, and also in the discussion a number of other food chemists took part. The discussion occupied two days and was quite complete. The committee recommends that the methods approved by the referee and associates, which are the circulars which have been distributed, with certain corrections that have been made owing to criticisms, be printed in a separate bulletin as provisional methods.

Adopted

The President. Has the abstract committee any report to make?

Mr. Allen. I have a very short report.

REPORT OF COMMITTEE ON ABSTRACTS.

Mr. President and Members of the Association: The committee on abstracts appointed by the incoming president included the members of the committee of the previous year so far as they desired to continue the work. Correspondence on the part of the chairman developed the fact that several members of last year's committee were not in position to keep up the work, and one other member had gone out of official work. Some difficulty was had in securing suitable recruits to the committee, and at the suggestion of the president of the association the number of members was reduced to five instead of nine, as formerly. This reduction in number, while it imposed a larger amount of work on the individual members of the committee, simplified the laying out of the work for the year and its management, as formerly no little trouble had been experienced in directing the work of the large and widely-scattered committee and keeping it up to date.

As in previous years, the work was divided by journals instead of by subjects, the former seeming to be the only practicable method, although it is recognized that an assignment by subjects might add to the interest of the individual members of the committee. The committee has attempted to follow up regularly twenty-seven of the most prominent journals containing articles on agricultural chemical methods, and has included, besides, abstracts from a long list of other journals which contain occasional articles in that field. The results of this work have been published as formerly in the Experiment Station Record from month to month during the year. Despite the reduction in the size of the committee, it is hoped that the scope and efficiency of the review has not suffered seriously.

E. W. Allen,
W. H. Beal,
C. B. Williams,
W. F. Hand,
D. W. May,
Committee.

Mr. Kilgore. Mr. Chairman, it was suggested by you in your address, I suppose upon considerations which came from the chairman of this committee, that the committee be discontinued. I would like to know if Mr. Allen is really of the opinion that the work could be done more efficiently by the regular editoral staff of the Record. If so, I think it would be well to adopt this report and discharge the committee. I do not think our men are looking for work that is really not necessary for them to do.

Mr. Allen. The conditions have changed somewhat from those that existed at the time the abstract committee was first suggested. For a number of years that committee aided quite materially in following up the literature relative to methods of agricultural analysis; but in the meantime the force in our office has grown considerably and has included among its additions a number of men who were familiar with the literature of agricultural chemistry and have followed up that literature. Now, in principle such a committee would seem to be of a great deal of assistance, and in the past that has been true. But tbe difficulty has been each year to get men who are willing to give their time to follow this literature regularly, and unless the work is done regularly a man soon falls behind and it becomes difficult to handle it, so that abstracts which treat the literature uniformly are rather difficult to obtain. I can understand this. I do not make it as any criticism on the members who have been appointed on the committee. Seasons come in the year when the work presses unusually hard in the laboratory, and if the work is allowed to lag it becomes more difficult to take it up. It has happened that after good men have been in this work for a year or two, until they have become thoroughly familiar with it, they have lost interest and wanted to be replaced by other members. The result has been that there has been a diminution in the number of the members and an attempt to get members who are nearer at hand here in Washington. The last year there were only two members of the committee we were able to obtain who were located outside of the Office of Experiment Stations. I feel now that we are in a position in the Office to follow up that literature with reasonable regularity and completeness. We are anxious, of course, in our own publications, to review all important articles relating to methods of agricultural analysis, and it becomes necessary, in order that we shall not overlook any important article, to open a ledger account with each man on the journals that are assigned to him, and that involves a good deal of work. Some man in the office must go over each number of this journal as they come in and make a note of those articles, which is kept as a matter of record; and if those articles are not abstracted in the course of a couple of months, he must undertake to abstract them. In that way we have sometimes delayed the abstracting, and it has made a great deal of work. So that while I appreciate very highly the work which the members of the committee have done, I believe now that the time has come when we can handle that work to the satisfaction of the association in the office.

Mr. Kilgore. We have approved the idea of expansion here this morning. To be on the safe side, I move that the association now approve the method of concentration, and that we accept this report and discharge the committee and concentrate the work in the Office of Experiment Stations.

The President. The Chair would suggest that you use the word "discontinue."

Mr. Kilgore. I accept the change.

The President. A motion has been made that the report be approved and the committee discontinued.

Carried.

The President. Has the committee on volumetric standards any report to make?

Mr. Kilgore. The committee has not had a meeting. The object for which this committee was appointed has been accomplished. It was to try to bring about a uniform standard with reference to the unit of volume as well as the graduation. As we all know, at the last Congress a bill was passed establishing a national standardizing bureau, and I think this association had great weight in bringing this about. I feel, therefore, that the work for which this committee was appointed has been done, and I should think that the committee might be discontinued.

Mr. Ewell. I would like to emphasize the fact that the work of this association and its members, particularly the chairman of the committee, who has just spoken, was very helpful indeed in bringing about this result, and I feel that the result and what the committee accomplished is an assurance that it is a good thing for scientists to tell legislative bodies what they want. It is not lobbying, in the accepted sense of the word. This is an excellent demonstration that Congress is ready to do what is wanted when the need is put before them in that light. This is the plan on which all the work of the standards bureau was done.

The PRESIDENT. Shall any action be taken on the suggestion of the chairman of the committee on volumetric standards?

Mr. WHEELER. I move its adoption and that the committee be discontinued.

Adopted.

The PRESIDENT. Mr. Frear, the chairman of the committee on food standards, has requested that this statement be read as a report of progress:

REPORT OF COMMITTEE ON FOOD STANDARDS.

Reports on the following subjects are ready for submission, presenting tentative food definitions and standards for criticism and suggestion before final adoption:

Meat products, lard, milk and milk products, flavoring extracts, spices, fruit preserves, etc., sugars and sirups, fruit juices, fermented liquors, tea, coffee, and cocoa preservatives.

Dr. J. B. Weems, of Iowa, has performed for the committee a considerable amount of research work in the study of the constants of the raw materials used in the manufacture of butter substitutes in America.

WM. FREAR, *Chairman.*

The report was adopted.

The PRESIDENT. We will now hear from the committee on resolutions. Mr. Wheeler has the floor.

REPORT OF COMMITTEE ON RESOLUTIONS.

The following resolution was presented by Mr. Ewell:

Resolved, That the title "Determination of nitrogen" be changed to read "Determination of nitrogen and nitrogenous bodies," and that the work on nitrogen be divided between two or more referees. Further, that the nitrogen methods be taken out of fertilizer methods and proper reference inserted in their place.

The committee recommend that this resolution be referred to the referee on nitrogen, with suggestions as to titles, etc., a definite report to be made at the next meeting of the association.

Mr. KILGORE. I move the adoption of the report of the committee, which will carry a reference of this to the nitrogen referee for a report at the next meeting.

Seconded.

Mr. EWELL. I move to amend by adding that the executive committee be instructed to consider the advisability of appointing two referees, one for methods for the determination of nitrogen, and the other for the separation of nitrogenous bodies for their determination, so that we may start the work between now and the next meeting.

Seconded.

The PRESIDENT. The amendment is that the executive committee be instructed to consider the subject of the appointment of referees in connection with the division of nitrogen work—that is, I suppose, a division on the lines laid down in the original motion.

Mr. Wheeler. I think that is perfectly satisfactory. The reason for laying over the general matter was that there might be an orderly and systematic consideration of the whole matter.

Amendment carried. Original motion as amended carried.

Mr. Wheeler. The next is a resolution introduced by Mr. Ross:

Resolved, That the president-elect appoint, during the future sittings of this convention, the committees on recommendations of referees who are to serve during the next succeeding annual convention.

The idea of this is that the men who are to serve on these committees will have notification and will be able to look up the matters upon which they are to pass judgment and come here better prepared to fill the positions than were they notified only after their arrival. I move the adoption of the resolution.

Carried.

The President. Has the committee on resolutions anything further?

Mr. Wheeler. That is all concerning resolutions. If it is in order, I would like to introduce a motion affecting the future policy of the association to a certain extent. I move that the committees on recommendations of referees shall meet during or at the close of the next annual convention and prepare their reports upon the recommendations of the referees, the same to be distributed to the members of the association at least six months before the next succeeding annual convention; provided, however, that by a two-thirds vote of the voting members present at any session the above-mentioned committee may be instructed to report during the same annual convention upon minor matters and matters of urgent necessity, and that action of the association may be taken thereon without delaying until the subsequent annual convention.

This provides that the association may, at the next annual convention, or at any annual convention, act upon matters of minor importance or of urgent necessity immediately; but in view of the fact that we assume to act upon a large number of new methods, particularly in connection with the food investigation, it was thought wise to make some provision by which all the proposed methods and all the important changes should be placed in the hands of every member of the association, so that he can look up the question and come here prepared to vote intelligently. I move that this be adopted.

Seconded.

Mr. Kilgore. Our constitution already provides that no matter shall be passed upon by the association with reference to the change in a method until it has been submitted to the test of the members of the association. It appears to me that this resolution concerns a matter which is fixed by the constitution, and I think that either the constitution ought to be changed so as to take in this matter, or else the resolution ought not to be passed.

The PRESIDENT. The constitution refers to those materials that are affected by inspection. It would be those materials that, as things stand now, would not be affected by this resolution.

Mr. WHEELER. This motion, as I understand it, is not in collision at all with that provision in accordance with which the methods are to be tested before adoption, but merely provides that all of the recommendations of the committee on recommendations of referees shall be distributed to each member of the association, so that, before the final vote is taken upon the adoption of these methods and upon important modifications, each member shall have seen the final report of these committees on the recommendations. We have seen to-day the fact that the body of the house is not in position to know just what these changes mean, because we have not the methods before us. I consider this is in nowise in conflict with the constitution. It only provides that every member shall be furnished with important recommendations.

Mr. KILGORE. I do not object to the resolution at all. The spirit and intent of it are good. But it would make a delay of two years before any change in a method could be brought about, instead of one year as provided now, and the constitution gives us the right to make a change within one year provided the test has been satisfactory and two-thirds of the association so vote.

Mr. WHEELER. I fear the motion is not understood. It was intended to provide that matters which arise next year, which under our constitution can not be adopted until the year following, may be adopted the year following just the same; but that before we come here to vote upon them we shall have a printed list of those recommendations. This does not delay the adoption, and provides that by a two-thirds vote we may act upon anything which is of immediate necessity—minor matters or matters of urgent necessity—and that all others will be acted upon just as early as they would be under our constitution.

Mr. HOPKINS. It seems to me that the machinery to accomplish these results is rather cumbersome. Would it not be possible to have the referee by circular letter notify the members of the association a month or so in advance of the meeting of the proposed changes, thus referring it, as you say, to a committee of the whole?

Mr. WHEELER. That plan has been considered by a number of the members of the association, and the trouble with that is that the recommendation of the referee is not the final recommendation upon which this association has to act. We are obliged to act upon the recommendation of the committee, and the referee is not in position to render any such report beforehand.

The PRESIDENT. I believe a motion has been made and seconded that the resolution as read be adopted.

Carried.

The PRESIDENT. Has Mr. Wiley any report to make for the committee on fertilizer legislation?

Mr. WILEY. The committee has not taken any action in regard to any fertilizer legislation, and it does not seem to me necessary to continue this committee any longer. If anyone thinks the committee should be continued of course it can be done, but it is hardly necessary to carry a committee unless there is something for it to do. The fact is that the fertilizer laws in the States are so different that it is almost impossible to draw any general scheme which will meet the approbation of any considerable number of States. If there could be a national fertilizer law regulating interstate commerce in fertilizers then the committee would have a raison d'être, because it could do some good work. Such a law would be desirable. But those of you who know the difficulty of even getting a national pure-food law may realize how remote the prospect is of getting one controlling interstate traffic in fertilizer materials.

It may be interesting for you to know that the President of the United States is interested in pure-food legislation to such an extent that he has asked the Secretary of Agriculture for an expression of opinion as to what steps the Executive should take in the matter, and it is more than likely that we shall have an expression from the President as to the advisability of Congress taking some action along this line.

I would like to hear from some of the gentlemen present who are directly in contact with State legislation as to the desirability of continuing this committee.

Mr. KILGORE. I feel that the committee should be continued. In the South we have been able to accomplish something. Our commissioners of agriculture of the cotton States have an organization, and through that organization we have drafted what we call a uniform fertilizer law for the States connected with the association. The legislature of the State of Georgia is now wrestling with that bill. The fact that this association has a committee on the subject has been helpful with us in getting the matter to the attention of the proper parties concerned, and for the moral effect of it at any rate we would like, in my section of the country, to see this committee continued.

Mr. WHEELER. I will say that a similar committee appointed by the Association of Agricultural Colleges and Experiment Stations has this year brought the matter before the United States commission on uniform legislation, and at their last meeting at Denver they instructed their members in the respective States to use their efforts, in cooperation with the committee of this association and the colleges and experiment stations, to secure modifications of laws in their respective States in accordance with this general scheme; and I feel that merely for the sake of keeping before our members the fact that there is a scheme after which it is hoped the laws of the individual States will be modeled, this committee should be continued.

Mr. Kilgore. I move that the report be approved and the committee continued.

Carried.

The President. I would say, in closing, that this has been in attendance the largest convention that the association has ever held. Last year the attendance was 85, which was the largest up to that time. This year the attendance is well on toward 100, and I think that the interested manifested in all of the sessions of the meeting, both by the number present and by the close attention given to the proceedings, indicate a state of thorough interest and support in the work of this association. In some respects this meeting has been an exceptional one aside from the large attendance. We have all noticed the comparatively small amount of time called for by discussions. This has been owing to the nature of the work which happened to be of a peculiar character this year. A large amount of new work came before the association which was in a measure pioneer in its character, and work which had to be largely considered and digested in committee outside of the convention's sessions. So that there has been a very much smaller amount of discussion than would have been called forth had we proceeded in the manner which we employed some years ago in bringing everything, even in embryo, onto the floor of the convention. For illustration, the committee on food adulteration, of which Mr. Bigelow was chairman, has been at work very busily this last year, and by the employment of the most admirable method which Mr. Bigelow used in getting the reports of the members of the committee printed and sent out for inspection and criticism, and then by having the associate referees of that committee hold meetings for the best part of two days here in Washington before and during this convention, this enormous mass of matter has been put before the convention in such a shape as to require a very small amount of time. Of course the time for discussion will properly come when these methods have been still further worked upon and digested and submitted to the future conventions of this association for final action. It seems to me that the association has made very great progress in the matter of economizing time. To those who have attended the convention of this association for the first time this year I would say, therefore, that this meeting has not been a typical one in every respect, but rather exceptional in point of the small amount of discussion, owing to the peculiar conditions that have prevailed, and in general to the comparative shortness of the session. It is not to be assumed, I trust, that our conventions hereafter will be necessarily as short. I am sure we can not be charged with any undue haste, nor can we be charged with undue delay in the proceedings.

I took pains in my opening address not to express any special gratitude to the association for having placed me in the position of presiding officer, for the reason that I felt that I could a good deal better and more truthfully express my feelings at the close of the convention than I might at the beginning. I would say now that I am grateful to the association for the ease with which they have made it possible for the chairman to preside at the sessions of the convention. The attention has been uniformly close and the behavior of the members has been remarkable.

Mr. KILGORE. I would like to offer this verbal resolution:

Resolved, That the authorities of the Columbian University have placed our association under renewed obligations to it by furnishing us facilities for our meeting, and that the sincere thanks of the association are extended to the authorities for this continuance of their favor.

I move the adoption of the resolution.

Carried.

Mr. WHEELER. I move that a hearty vote of thanks on behalf of the association be extended to the Cosmos Club for their courtesy, and a similar vote of thanks to the Secretary of Agriculture for his patronage and assistance by which we have been able to have the reports here for distribution; and that the secretary be instructed to transmit to the secretary of the Cosmos Club and to the Secretary of Agriculture the expression of the thanks of this association.

Mr. KILGORE. And I would like to have included in the first resolution that the secretary furnish a copy to the authorities of Columbian University.

Mr. WHEELER. I am glad to include that in my motion.

Carried.

Mr. ROSS. I move that the thanks of this association be tendered to the retiring president for the able, impartial, and courteous manner in which he has presided over its deliberations.

Carried.

At 11.15 a. m. the convention adjourned sine die.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1902.

President.

Mr. H. J. Wheeler, Kingston, R. I.

Vice-President.

Mr. R. J. Davidson, Blacksburg, Va.

Secretary.

Mr. H. W. Wiley, Washington, D. C.

Additional Members of Executive Committee.

Mr. C. G. Hopkins, Champaign, Ill.
Mr. F. D. Fuller, Geneva, N. Y.

Referees.

Phosphoric acid: C. H. Jones, Burlington, Vt.
Nitrogen:
    Determination of nitrogen: Fred W. Morse, Durham, N. H.
    Separation of nitrogenous bodies: L. L. Van Slyke, Geneva, N. Y.
Potash: H. B. McDonnell, College Park, Md.
Soils: F. P. Veitch, Washington, D. C.
Dairy products: George Cavanaugh, Ithaca, N. Y.
Foods and feeding stuffs: C. A. Brown, jr., State College, Pa.
Food adulteration: W. D. Bigelow, Washington, D. C.
Sugar: G. L. Spencer, Washington, D. C.
Tannin: Wm. H. Teas, Ridgway, Pa.
Insecticides: J. K. Haywood, Washington, D. C.
Ash: G. S. Fraps, Raleigh, N. C.

Associate Referees.

Phosphoric acid: B. H. Hite, Morgantown, W. Va.
Nitrogen:
    Determination of nitrogen: Edward B. Holland, Amherst, Mass.
    Separation of nitrogenous bodies: R. Harcourt, Guelph, Canada.
Potash: Henri D. Haskins, Amherst, Mass.
Soils: C. C. Moore, Washington, D. C.
Dairy products: C. A. Brown, jr., State College, Pa.
Foods and feeding stuffs: F. D. Fuller, Geneva, N. Y.

Food adulteration:

(1) Meat and fish: W. D. Bigelow, Washington, D. C.
(2) Fats and oils, not including dairy products or their substitutes: L. M. Tolman, Washington, D. C.
(3) Cereal products: A. McGill, Ottawa, Canada.
(4) Infants' and invalids' foods: H. W. Wiley, Washington, D. C.
(5) Saccharine products, including confectionery: A. E. Leach, Boston, Mass.
(6) Vegetables—canned, dried, or otherwise preserved: L. S. Munson, Washington.
(7) Tea and coffee: W. H. Ellis, Toronto, Canada.
(8) Cocoa: F. T. Harrison, London, Ontario.
(9) Spices and condiments: A. L. Winton, New Haven, Conn.
(10) Vinegar: Wm. Frear, State College, Pa.
(11) Flavoring extracts: A. S. Mitchell, Milwaukee, Wis.
(12) Fruit products: L. M. Tolman and L. S. Munson, Washington, D. C.
(13) Fermented and distilled liquors: W. D. Bigelow, Washington, D. C.
(14) Baking powders nd baking powder chemicals: A. L. Winton, New Haven, Conn.
(15) Preservatives: W. M. Allen, Raleigh, N. C.
(16) Dyes: L. M. Tolman, Washington, D. C.

Sugar:

Optical methods: L. M. Tolman, Washington, D. C.
Chemical methods: L. S. Munson, Washington, D. C.
Special analytical methods used in sugar industry: D. S. Davol, Caro, Mich.

Tannin: Geo. A. Kerr, Damascus, Va.
Insecticides: James Emory, Washington, D. C.
Ash: F. T. Shutt, Ottawa, Canada.

COMMITTEE ON FOOD STANDARDS.

Mr. Wm. Frear, State College, Pa., Chairman.
Mr. H. W. Wiley, Washington, D. C.
Mr. H. A. Weber, Columbus, Ohio.
Mr. M. A. Scovell, Lexington, Ky.
Mr. E. H. Jenkins, New Haven, Conn.

COMMITTEE ON FERTILIZER LEGISLATION.

Mr. H. W. Wiley, Washington, D. C., Chairman.
Mr. B. W. Kilgore, Raleigh, N. C.
Mr. H. B. McDonnell, College Park, Md.
Mr. H. A. Huston, Lafayette, Ind.
Mr. B. B. Ross, Auburn, Ala.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists of the United States. The objects of the association shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership; and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to enter motions or vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. All persons eligible to membership shall become members ex officiis and shall be allowed the privileges of membership at any meeting of the association after presenting proper credentials. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to have all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall not be entitled to enter motions or vote.

(3) The officers of the association shall consist of a president, a vice-president, and a secretary, who shall also act as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee, to continue in office till the annual meeting next following.

(4) There shall be appointed by the executive committee, at the regular annual meeting, a referee and such associate referees for each of the subjects to be considered by the association as that committee may deem appropriate.

It shall be the duty of these referees to prepare and distribute samples and standard reagents to members of the association and others desiring the same, to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) No changes shall be made in the methods of analysis used in official inspection, except by unanimous consent, until an opportunity shall have been given all official chemists having charge of the particular inspection affected to test the proposed changes.

(8) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(9) The executive committee will confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(10) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by the approval of two-thirds of the members present entitled to vote.

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